

THERMAL COOK-OFF STUDY OF PRESSED NITROTRIAZOLONE (NTO)  
PELLETS

A Thesis

by

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## ABSTRACT

As of today, no research has been performed to study the response of Compounded High Explosive Composite (CHEC) pellet arrays to fast or slow cook-off. Cook-off tests are fairly standard tests for homogeneous-fill high explosives (HE's), but, when using pressed pellet arrays as fill, the results of such cook-off tests are unknown. This research consisted of developing and designing an “indoor” cook-off test capable of testing pressed Nitrotriazolone (NTO) pellets to assess thermal stability characteristics and critical temperature.

The test method designed consists of the main testing vessel—the Petersen Group strand burner IV composed of 17-4 PH stainless steel with extreme pressure and temperature capabilities—a sample bolt holder used to hold the NTO pellet, a 400-W cartridge heater, two type K thermocouples, and an aluminized heat barrier. The cartridge heater was operated using a proportional-integral-derivative (PID) temperature controller. The data from the thermocouples used to monitor pellet and surrounding air temperatures were recorded using a USB-interfaced data logger capable of taking measurements at a rate of 1 sample per second.

A series of thermal cook-off tests was conducted revealing critical temperatures for the NTO pellets ranging between 175 and 203°C. For slow cook-off tests, the average critical temperature,  $T_C$ , was determined to be  $196 \pm 7^\circ\text{C}$ . Fast cook-off tests revealed an average  $T_C$  of  $189 \pm 1^\circ\text{C}$ . Due to these temperatures occurring over such a wide range and evidence of partially burned explosive residue material after each test, it

is suggested that the pellets are undergoing premature decomposition, and thus a critical temperature for these NTO pellets cannot be determined to a high accuracy. As such, the thermal stability of NTO pellets in CHEC arrays remains to be verified. This thesis details the design and development of the “indoor” cook-off test as well as the thermal characteristics of the NTO pellets when exposed to extreme heating.

## DEDICATION

I dedicate this thesis to my parents, Michael and Jennifer: my inspirations and my heroes. Their constant support and encouragement, high expectations and demand for excellence, and undying love, are what have helped shape me into the young woman I am today. Words cannot express how thankful I am to them for all of the sacrifices they have made for me, throughout my entire life. It is because of them that I have been able to achieve my dreams. I am so incredibly blessed to call them my parents. I love you both bunches and bunches, forever and always!

*“I always thank God for you because of his grace given you in Christ Jesus. For in him you have been enriched in every way—in all your speaking and in all your knowledge...”*

*1 Corinthians 1:4-5*



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Thank you to the Naval Surface Warfare Center: Indian Head Explosive Ordnance Disposal Technology Division (NSWC IHEODTD) for supporting me in my research efforts. A sincere thank you to my boss, Adam Goldberg, for taking the extra time and effort to ensure that my research was successful, and ensuring that I was provided with all the knowledge, guidance, and materials necessary. I would also like to thank my mentor, Mary Sherlock, for her advisement and guidance through the entire research process.

Being as the world of explosives research and testing is fairly small, I would also like to thank those from whom I sought additional knowledge/advice: Dr. John Meason, former Director of the Energetic Materials Research and Testing Center (EMRTC), Dr. Michael Hargather of the New Mexico Institute of Mining and Technology (New Mexico Tech, NMT), Paul Giannuzzi of NSWC IHEODTD, Fred Sandstrom of Applied Research Associates, Inc., and Melissa Lyons of K2 Construction Consultants, Inc.—all

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## CONTRIBUTORS AND FUNDING SOURCES

### *Contributors*

The work of this thesis was overseen by the student's thesis committee: Dr. Eric Petersen (chair) and Dr. Waruna Kulatilaka (member) of the Department of Mechanical Engineering, and Dr. Chad Mashuga (member) of the Department of Chemical Engineering. Additional advisement was provided by Mary Sherlock of the Naval Surface Warfare Center: Indian Head Explosive Ordnance Disposal Technology Division (NSWC IHEODTD), R12 Organization.

All work completed for this thesis was done so by Michaela Fasano under the direct advisement of Dr. Eric Petersen.

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The contents of this report are solely the responsibility of the author and do not necessarily reflect upon the official views of the NSWC IHEODTD.

## NOMENCLATURE

CHEC	Compounded High Explosive Composite
$D_{cr}$	Critical Thickness
DoD	Department of Defense, U.S.
DoE	Department of Energy, U.S.
DSC	Differential Scanning Calorimeter
EM	Energetic Material
ESD	Electrostatic Discharge
H	Height
HE	High Explosive
HMX	Cyclotetramethylenetetranitramine
IHEODTD	Indian Head Explosive Ordnance Disposal Technology Division
IM	Insensitive Munition
LE	Low Explosive
MSDS	Material Safety Data Sheet
NSWC	Naval Surface Warfare Center
NTO	3-Nitro-1,2,4-Triazol-5-one (or Nitrotriazolone)
PBX	Plastic-bonded Explosive
PETN	Pentaerythritol Tetranitrate
PID	Proportional-Integral-Derivative
RDX	Cyclotrimethylene Trinitramine

SB-IV	Strand Burner IV
SSCB	Super Small Cook-off Bomb
T <sub>C</sub>	Critical Temperature
TMD	Theoretical Maximum Density
VTs	Vacuum Thermal Stability
W	Width

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## CHAPTER I

### INTRODUCTION

#### **Energetic Materials**

An energetic material (EM), also referred to as an explosive, is any material capable of violent or explosive decomposition when subjected to friction, impact, static electricity, or heat. The United States Department of Defense (DoD) defines explosives as an “energetic substance (or a mixture of substances) which is in itself capable, by chemical reaction, of producing gas at such temperature, pressure, and speed as to cause damage to the surroundings.”<sup>1</sup> This term refers to all solid and liquid energetic materials: high explosives and propellants together with igniter, primer, initiator, and pyrotechnic compositions.<sup>1</sup>

Explosives are categorized into two main groups: high and low explosives. Low explosives (LE), which are further broken down into gas-producing (propellants, black powder, primer and igniter mixtures) and non-gas-producing (gasless type delay composition), are explosives that deflagrate or burn.<sup>2,3</sup> This definition is due to how the rate of advance of the chemical reaction zone into the unreacted explosive material is less than the velocity of sound through the material. In contrast, high explosives (HE) detonate rather than deflagrating or burning because of how the chemical reaction zone advances at a rate greater than the velocity of sound in the unreacted material. HEs are also further divided into three groups: primary explosives (high sensitivity and easy

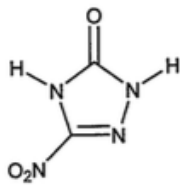
detonability), secondary explosives (relatively insensitive), and tertiary explosives (high insensitivity).<sup>2</sup>

This research focuses on the use of Nitrotriazolone (NTO), a secondary explosive, also referred to as an insensitive munition (IM). According to the DoD Test Method Standard, IMs are “munitions which reliably fulfill (specified) performance, readiness, and operational requirements on demand but which minimize the probability of inadvertent initiation and severity of subsequent collateral damage to the weapon platforms, logistic systems, and personnel when subjected to unplanned stimuli.”<sup>1</sup> In other words, IMs are meant to respond less violently to accidental environmental stimuli. The development of IMs came about as a result of accidents caused by explosives; these incidents drove the military to develop new energetics with drastically reduced sensitivity that could replace the explosives being used in warheads at the time. NTO was one of these explosives developed as a solution.

### **Nitrotriazolone (NTO)**

3-Nitro-1,2,4-triazol-5-one (NTO) is an insensitive high explosive that exhibits similar energetic performance as cyclotrimethylene trinitramine (RDX) with improved sensitivity to shock, impact, and heat. Table 1 provides a brief overview of the chemical properties of NTO.

**Table 1:** Overview of chemical properties of NTO taken directly from Smith and Cliff<sup>4</sup> and the Ordnance Systems Material Safety Data Sheet<sup>5</sup>.

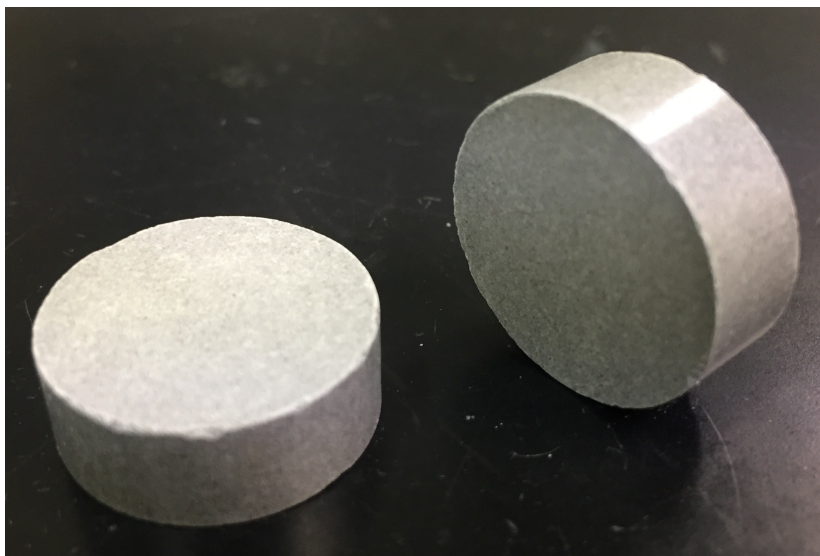
Chemical Properties of NTO	
Common Names	3-nitro-1,2,4-triazol-5-one (NTO) 5-oxy-3-nitro-1,2,4-triazole (ONTA) 5-nitro-2,4-dihydro-3H-1,2,4-triazol-3-one
Empirical Formula	C <sub>2</sub> H <sub>2</sub> N <sub>4</sub> O <sub>3</sub>
Structure	
Molecular Weight	120.0646 g/mol
Oxygen Balance	-24.60%
Appearance and Odor	Light green to white solid; No odor.

For this study, NTO was pressed into 0.75-in diameter by 0.375-in height pellets by the Naval Surface Warfare Center Indian Head Division, late Yorktown Detachment. A series of small-scale safety testing was performed on the NTO material to ensure its stability for pressing operations. Such testing provided significant differential scanning calorimetry (DSC), vacuum thermal stability (VTS), friction, impact, and electrostatic discharge (ESD) information for the specific lot of material. A Stokes Rotary Press was used to press the pellets. The NTO powder was pressed three times at 5 kpsi pressure intervals up to a maximum of 30 kpsi without vacuum. The increasing pressure increments corresponded with an increase in pellet density up to 1.79 g/cc at 20-30 kpsi, approximately 93% of the theoretical maximum density (TMD) of 1.93 g/cc. However, due to the presence of striations and cracking within the pellets as a result of trapped air, a combination of 0.5% calcium stearate and 0.5% graphite was added. Graphite is

frequently used in molding powders and mechanically blended into explosive powders prior to pressing to aid in lubrication and processability of the powder, and calcium stearate helps to bond the powder particles together with the graphite. These additions stopped crack formation, with all pellet densities still exceeding the 91% TMD requirements.<sup>6</sup> The following table (Table 2) presents the sensitivity and thermal data for the actual NTO pellets used in this research<sup>7</sup>, followed by Figure 1 which shows the actual pellets used for testing.

**Table 2:** NTO Pellet Laboratory Sensitivity Results from research conducted by the Yorktown Detachment of the Indian Head Naval Surface Warfare Center.<sup>7</sup>

NTO Sensitivity Data		
	NTO	Reference Value
DSC	269.18°C	
VTS	0.07 ml/g	
Friction	360 N	PETN: 48 N
Impact Sensitivity	182 cm	RDX: 14.6 cm



**Figure 1:** NTO pellet samples.

### **Compounded High Explosive Composite (CHEC) Technology**

Traditionally, to reduce the sensitivity of high explosives, less-sensitive explosive powders, oxidizers, and fuels have been substituted for more-sensitive high explosive powders such as RDX and HMX. Consequently, the critical diameter of these less-sensitive formulations drastically increases, requiring a larger booster, which as a result makes it more vulnerable to external threats. The critical diameter of an explosive is the minimum diameter at which a steady-state detonation will propagate.<sup>8</sup> Thus, if the diameter of the explosive charge is reduced by an extremely small amount, then the diameter of the charge will equal the failure diameter, and the detonation fails to occur.

An alternative approach to reducing weapons hazards —i.e. Compounded High Explosive Composite (CHEC) Technology described in U.S. Patent 8,943,971<sup>9</sup>— involved arranging explosive pellets in such a manner so as to decrease sensitivity to radial stimuli while maintaining sufficient axial sensitivity for initiation by a standard booster. CHEC is a U.S. military high explosive (HE) technology only and is not meant for propellants.

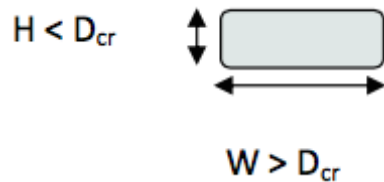
Compounded High Explosive Composite (CHEC) warhead structures provide a directionally sensitive structure for unprecedented control to allow for a steady state detonation of the high explosive along the warhead's axial boosting pathway. This controllability ensures proper warhead functioning while providing an unstable configuration that disrupts and dissipates wave propagation from the known vulnerability of radial (side) impacts. The sides of the warhead constitute the greatest area for attack as missile warheads are mainly shielded on their ends.

Patent 8,943,971 describes a novel method of assembling and filling HEs to limit their susceptibility to impact-initiated detonation. Existing warheads contain a homogeneous HE fill and thus can be detonated with the same shock input from any direction, whether intentionally detonated as designed (by the warhead fuze booster), or inadvertently by combat hazards such as stray fragments, bullets, and blast. Patented CHEC technology provides a novel family of low-cost explosives that exhibit anisotropic (directionally dependent, non-symmetric) sensitivity properties. CHEC exploits HE bulk properties through precisely tuned HE pellet arrays that cooperate to promptly and reliably detonate a warhead as designed, while greatly diminishing any possibility of initiation by external impact hazards or hardened targets. The effective energy content of the CHEC super-structure is equivalent to a conventional gravity-cast, plastic-bonded explosive (PBX), but the detonation characteristics are completely different—and controllable. Energy density scaling laws require these pressed HE pellets (92-96% solid energetic material (EM) by weight) to be heterogeneously arranged so as to provide necessary anisotropic sensitivity while mimicking the energy density of the gravity cast-cured homogeneous explosive (82-88% solid EM by weight). CHEC pellet architectures can be used to develop EM's and warhead superstructures that incorporate discrete pieces of functionality and anisotropic sensitivity properties making them independently scalable, similar to LEGO® building blocks.

CHEC consists of assemblies of small, highly consolidated explosive units arranged in an energy-dampening rubbery matrix. These HE sub-units are finely tuned to the specified HE bulk properties and are spatially distributed to form a super structure.



Bulk explosive charges can only sustain a detonation when their transverse dimensions are sufficiently large and supercritical. As a result, when these conditions are not met, incipient detonations fail because of rarefactions that encroach upon their reaction zones. As such, these individual HE unit cells are designed so that they are too small to sustain or propagate a detonation in the direction most vulnerable to threats: radially. Figure 2 illustrates basic notional scaling laws for the HE sub-unit as related to the critical thickness of the HE bulk explosive properties, where  $H$  is the height of the pellet and  $W$  the width. The critical thickness,  $D_{cr}$  (similar to critical diameter) is the minimum thickness in which an explosive can sustain a steady state detonation wave in a cylindrical charge, regardless of the volume of explosive.



**Figure 2:** Scaling laws used for NTO pellets, taken directly from US Patent 8,943,971.<sup>9</sup>

Now, the unit cell scaling is such that the impact stimulus across the thin radial cross-section is too weak to initiate detonation in a single discrete cell. Localized ignition will most likely occur due to deformation and shear; however, steady state propagation along the radial direction is inhibited. Typical cast-cured PBX compositions have failure diameters well below 0.5 in; large-critical-diameter explosives used in IM can have critical diameters an order of magnitude larger. Due to the scaling, CHEC formulations incorporated the modern insensitive explosive NTO. NTO's insensitivity

and large critical diameter makes the manipulation of its properties within HE subunit geometries possible.

### **Objective**

The technical objective of this research was to study the effects of thermal cook-off of pressed NTO pellets at various heating rates. The CHEC application of these NTO pellets in warheads has already been proven effective in limiting a warhead's susceptibility to impact-initiated detonation caused by various external threats. However, the determination of whether or not these pellet arrays possess different thermal stability characteristics than their homogeneous counterparts has yet to be discovered. By conducting a series of cook-off tests, the critical temperature of these pellets will be able to be determined and compared to values reported in the literature. This information will ultimately reveal if the use of CHEC NTO pellet arrays provides increased or decreased thermal stability properties upon potential exposure to extreme heating, such as in the case of an aircraft or vehicle fire.

Chapter II provides a literature review of the thermal cook-off of high explosives and the thermal decomposition of NTO. This is followed by an experimental analysis predicting pellet performance parameters in Chapter III, which also includes a complete overview of the development and design of the “indoor” cook-off test. Chapter IV reviews cook-off testing procedures and experimental results. This thesis concludes with an overview of the challenges associated with this research, as well as recommendations for further experiment optimization.

## CHAPTER II

### LITERATURE REVIEW

#### **Thermal Cook-off of High Explosives**

Extensive research has been conducted, mainly by United States Government institutions including the Department of Defense (DoD) and Department of Energy (DoE), to study the response of confined and unconfined energetic materials to ignition via bulk heating, i.e. cook-off, from an external source. Understanding such behavior is significant from a safety point of view, as explosives are not meant to be heated to extreme temperatures, especially in the case of slow cook-off where the material is allowed to undergo significant chemical and physical changes.<sup>10</sup>

A variety of thermal tests can be performed on explosive materials, including NTO, to characterize their response to various stimuli encountered both in storage and in the field. These tests are designed to closely reproduce real-life conditions to better understand the associated hazards. Small-scale and large-scale cook-off tests were developed by which the fundamental thermochemical and kinetic characteristics of the material or mixture can be determined.<sup>11</sup>

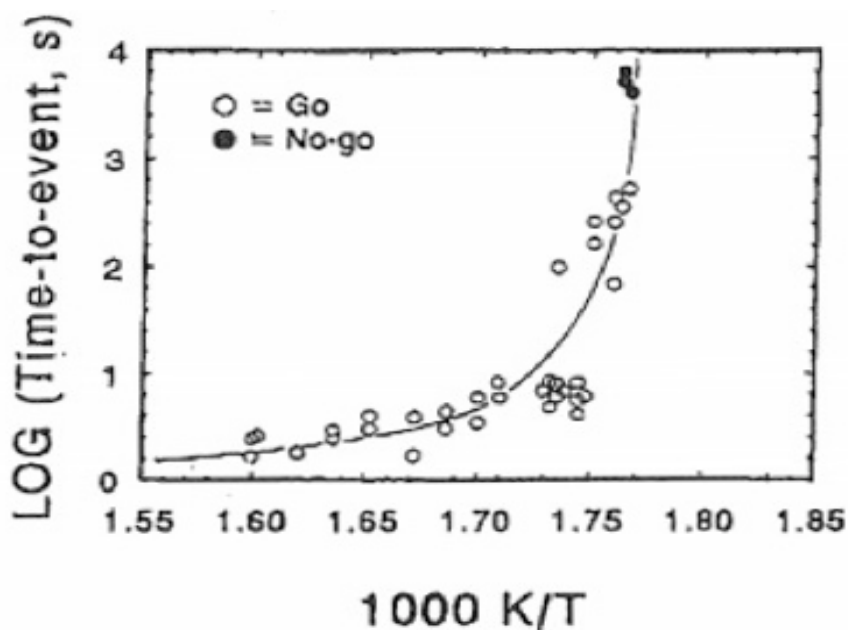
Cook-off, by definition, is the “deflagration or detonation of explosive material by the absorption of heat from its environment”<sup>2</sup> whose process involves the combined effects of different physical and chemical processes including heat transfer, chemical decomposition and mechanical response.<sup>12</sup> To achieve ignition or detonation, the explosive sample must reach its critical temperature ( $T_C$ ). As energetic materials

decompose, they will generate heat. As the temperature increases, the rates of decomposition and heat generation increase. Therefore, when the sample reaches a temperature in which it can no longer dissipate heat faster than it is being generated, the sample will go into a self-sustaining, thermal runaway reaction. The temperature where this occurs is referred to as the critical temperature ( $T_C$ ).<sup>13</sup> It is important to note, however, that the critical temperature is not a constant value, but a property that varies with the size of the explosive charge, the surrounding environment thermal properties, and time of exposure.<sup>2</sup>

There are two types of cook-off tests: fast and slow. Fast cook-off, which is characterized by its high heat fluxes, is the process of rapidly heating an energetic material. Due to the high heating rate, only the outer edges of the sample are significantly heated; therefore, ignition occurs on the outer surface while the majority of the material is still at ambient conditions. In this case, the charge will most likely detonate rather than deflagrate.<sup>14</sup> Slow cook-off, also referred to as an isothermal cook-off test, is a hazards assessment test to assess the response of a munition exposed to a continuous, low heating rate (3.3°C per hour). Ignition typically takes place within the bulk of the energetic material in this case, most likely deflagrating rather than detonating.<sup>14</sup>

Due to the safety risks associated with cook-off testing in general, two standard tests were developed to provide critical information regarding the thermal runaway of a HE (especially in the case of newly created energetic materials where behavior is unknown): the Henkin-McGill test and the Navy Small-scale Cook-off Bomb (SCB).

The Henkin-McGill<sup>11,15</sup> test is a laboratory-scale test that utilizes a 40-mg sample of a solid explosive pressed into an aluminum blasting cap shell that is sealed with an aluminum plug. Aluminum has a high thermal conductivity and therefore provides even heating of the sample throughout. The sample is then immersed in a temperature-controlled, molten metal bath that is remotely actuated and operated. Typical results of this test include shell rupture for high explosives; however, some materials will exhibit a weaker “pop” when dislodging the sealing plug. Immediately, the time to explosion is measured and considered complete only when the sound created via shell rupture or plug disengagement is heard. Thus, in this case, the critical temperature is defined as the minimum temperature at which the sample—of specific size and shape—can be heated without undergoing thermal runaway or explosion. Sample Henkin-McGill test data for a castable emulsion explosive may be seen in Figure 3.

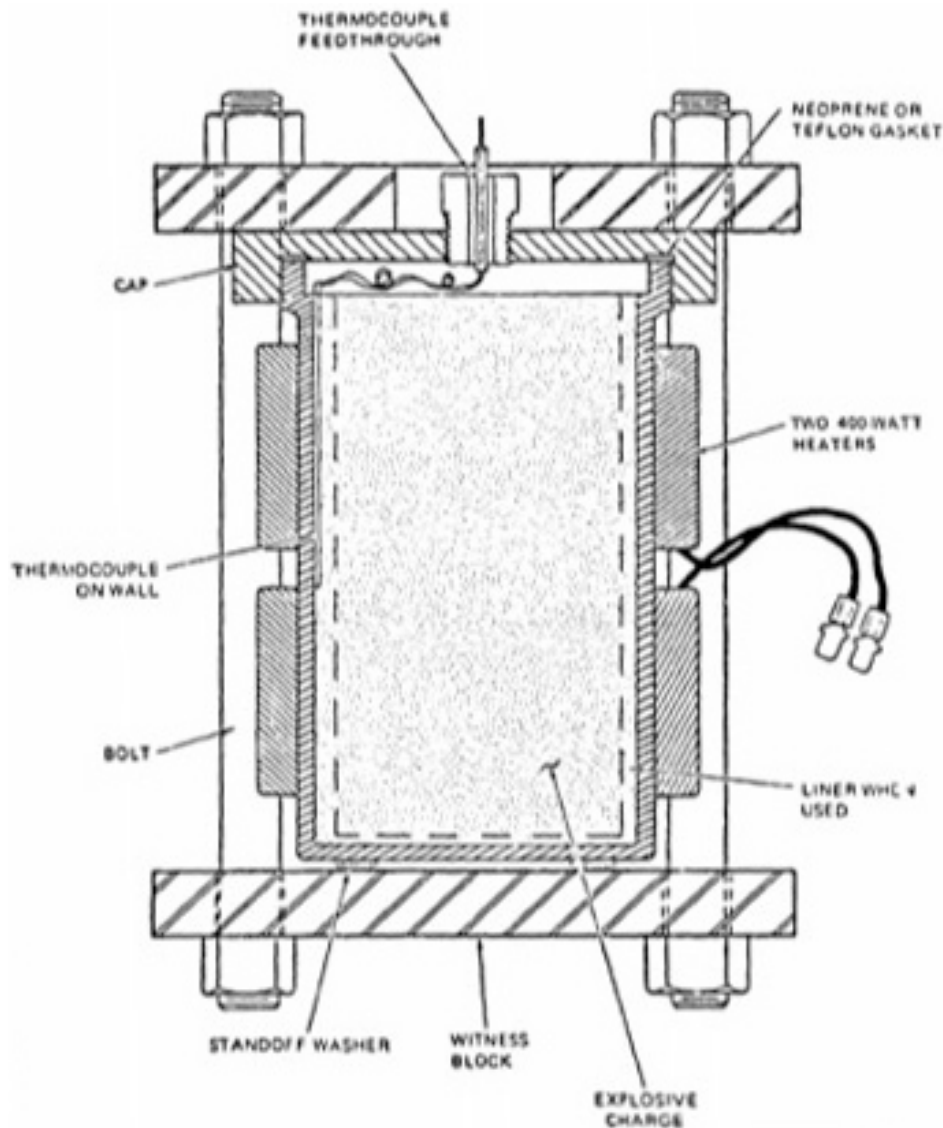


**Figure 3:** Henkin-McGill test data for a castable emulsion explosive taken directly from Olson and Banks.<sup>11</sup>

The Henkin-McGill critical temperature of NTO is reported to be  $226.5 \pm 10.5^\circ\text{C}$ .<sup>16,4</sup> This temperature most closely resembles that of RDX ( $219.6^\circ\text{C}$ ) and HMX ( $210^\circ\text{C}$ ).<sup>4</sup>

Small-scale Cook-off Bomb is used for tests of a slightly larger scale ( $\sim 20\text{ g}$ )<sup>17,15</sup> and provides not only the critical temperature of the EM, but also assesses the severity of the explosive material's response to thermal cook-off by examining the damage imposed to the SCB case and witness plate. This test requires the explosive material be confined in a steel cylindrical vessel with 3-mm thick walls, where heating is effected via two electric heater bands whose heating rate can be varied by varying the voltage applied to the heater.<sup>18</sup> An aluminum liner is placed inside the outer cylinder, around the explosive charge, to act as a heat sink to provide more-even heating throughout. A thin standoff washer is also placed below the aluminum liner and explosive material to act as an air-gap between the HE and the baseplate.<sup>17</sup> The vessel is then clamped between two steel witness plates (1.27 cm thick) using four bolts. The device is equipped with two plate-type thermocouples. In the case of a single HE being tested, a thermocouple is spot welded to the inside center of the vessel wall; for two HE's being tested simultaneously (i.e. a liner material plus the HE), a second thermocouple is placed between the two materials.<sup>18</sup>

The explosive fill inside the vessel must sit about 1 cm from the top of the cylinder to allow for thermal expansion. The types of explosive materials that may be tested using the SCB include solid, liquid, slurry, powder, or even gas under a modified assembly and fill conditions. A schematic of the SCB assembly is seen in Figure 4.



**Figure 4:** Small-scale Cook-off Bomb (SCB) schematic, taken directly from Pakulak.<sup>18</sup>

This test is typically conducted inside a firing chamber equipped with remote operations to control heater temperature and record thermocouple data.

For both of these testing methods, several tests are performed to determine an accurate minimum thermal runaway temperature, i.e. the critical temperature, for a given charge size. It is common for cook-off tests to first be performed using a slow heating

rate, building up to a fast heating rate, to determine the material's response over a specified temperature range.<sup>11</sup>

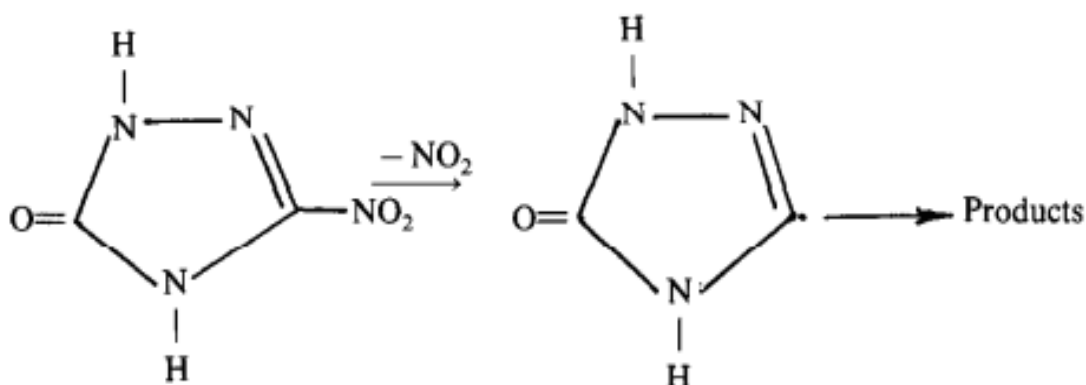
### **Thermal Decomposition of NTO**

3-Nitro-1,2,4-triazol-5-one (NTO), recognized as a high-energy material with high intensity and high brisance, is known for its ability to withstand very high thermal shocks. Various studies of the thermal decomposition of NTO have been performed, all of which focus mainly on three areas<sup>4</sup>: bond homolysis, nitro-nitrate rearrangement, and rupture of the triazole ring.

Prabhakaran et al.<sup>19</sup> conducted a bond hemolysis study to understand the relationship between the thermal stability of NTO compounds and their molecular structure by determining the complex reaction pathways in its thermal decomposition under optimized experimental conditions. Via curves obtained using thermogravimetry (TG) and differential thermal analysis (DTA), NTO is found to undergo decomposition in two stages: between 251°C to 262°C, and above 262°C, the first stage progressing more rapidly than the second. Such tests also reveal how NTO decomposes without melting. Specific tests of NTO in pellet form indicate a deceleration in decomposition, which perhaps suggests a diffusional resistance to the product gases—carbon dioxide (CO<sub>2</sub>), nitrous oxide (N<sub>2</sub>O), nitrogen dioxide (NO<sub>2</sub>), and nitric oxide (NO)—escaping from the reaction interface.<sup>19</sup> Thorough analysis of the decomposition behavior of NTO further suggests the breaking away of the C—NO<sub>2</sub> group weakens and ruptures the



adjacent C—N bond giving rise to the formation of the amide group (derived from carboxylic acids, containing the  $\text{—CONH}_2$  group) as seen in Figure 5.



**Figure 5:** Decomposition of NTO, taken directly from Prabhakaran.<sup>19</sup>

In contrast, the nitro-nitrite rearrangement study<sup>4</sup>, which utilizes laser ionization mass spectrometry diagnostics, provides evidence of the loss of NO rather than  $\text{NO}_2$ . In this study, the rearrangement of the nitro group to a nitrite is shown to be followed by the loss of NO and finally the rupture of the nitrogen ring ultimately suggesting several decomposition pathways.<sup>16</sup> As for the rupture of the triazole ring, this method of study<sup>4</sup> indicates a different phenomenon in that the thermal decomposition of NTO occurs via the intermolecular oxidation by the nitro group at the keto carbon atom<sup>16</sup> causing an appreciable loss of  $\text{CO}_2$  followed by  $\text{N}_2$ , detecting no  $\text{NO}_2$ . However, a separate study<sup>20</sup> that also confirmed the breakdown of the azole ring, did in fact find evidence of the elimination of  $\text{NO}_2$  like Prabhakaran<sup>19</sup>.

All in all, a considerable number of thermal decomposition studies of NTO have been performed, but there remains no assertive study regarding the exact mechanism of the process.<sup>16</sup> As a result, the kinetic data reported from such studies vary considerably.

For NTO, the reported autoignition temperature and heat of combustion are reasonably consistent; however, values for the heat of formation, activation energy, and heat of explosion vary. Table 3 provides the ranges of values in which such data have been reported:

**Table 3:** Thermal properties of NTO from Smith and Cliff.<sup>4</sup>

Thermal Characteristics of NTO	
Heat of Formation	-59.7 to -129.4 kJ/mol
Heat of Explosion	-392.7 to -602.2 kJ/mol
Heat of Combustion	-943.4 to -995.7 kJ/mol
Specific Energy	119.0 kJ/mol
Activation Energy	503.4 to 520.3 kJ/mol *This value has been reported as low as 170.1 kJ/mol, suggesting it to be markedly affected by sample and experimental conditions.
Autoignition Temperature	529.6 K = 256.5°C

## CHAPTER III

### EXPERIMENTAL DESIGN

#### **“Indoor” Cook-off Test Design Requirements**

As with all other forms of testing, a major concern of experimenting with high explosives is safety. For this research, the primary task consisted of developing and designing a safe “indoor” cook-off test that could be performed at Texas A&M’s Turbomachinery Laboratory. While the sample sizes of NTO tested were small scale, the fact that standard cook-off tests are almost always performed at an outdoor test range or in a large-scale boom box required a thorough analysis of pellet behavior when exposed to extreme temperatures. This entailed a complete and accurate assessment of the pressures the heated pellet would generate to determine the type of apparatus required to withstand such threats. Based upon the analysis performed in the following section, the Petersen Group strand burner IV (SB-IV) was chosen for conducting such cook-off tests.

#### **Engineering Analysis**

Thermal cook-off tests were performed inside the current Petersen Group SB-IV based upon its very high temperature and pressure capabilities. The SB-IV consists of a main body, two end-caps and a bolt-style sample holder, all machined from 17-4 PH stainless steel which provides significantly high yield and tensile strengths, as well as high-temperature compatibility. The analysis of the entire design of the SB-IV may be referenced in the Dillier thesis report: “Development and Characterization of a New

Very High-Pressure Strand Burner for Studying Propellant Burning Rates at Extreme Temperatures.”<sup>21</sup> Prior to performing the actual cook-off tests, an in-depth analysis of the behavioral characteristics of NTO pellets was conducted. Characteristics assessed included the thermal conductivity of the NTO pellets, the critical temperature of the pellet material, an approximation of the total pressure generated by the pellets due to extreme heating, and the estimated time to heat each sample to the critical temperature.

The thermal conductivity of the pressed NTO pellets was determined using the following conduction relationship (Equation 1)<sup>22</sup>,

$$\dot{Q}_{cond} = 2kA \frac{\Delta T}{\Delta x} \quad (1)$$

where  $\dot{Q}_{cond}$  is the rate of heat conduction radiated by the cartridge heater through the air to the pellet [W],  $k$  is the thermal conductivity [W/m•K],  $A$  is the surface area [m<sup>2</sup>],  $\Delta T$  the change in temperature [K], and  $\Delta x$  the thickness [m]. To determine  $\dot{Q}_{cond}$ , the system was set up<sup>23</sup> so as to assess the heat transfer through the fluid (i.e. air) layer that exists between the cartridge heater and pellet. In this case, the heat transfer through the layer is by conduction as the fluid layer is motionless. Using the 400-W rating of the cartridge heater to compute the actual heat emitted from the surface of the heater, the value of  $\dot{Q}_{cond}$  was found to be 15.06 W. Evaluating Equation 1 for  $k$  of the NTO pellets heated from room temperature (approximately 25°C) to a subcritical, uniform temperature of 160°C, the thermal conductivity was determined to be 0.9298 W/m•K. It is important to note that the majority of HE's have rather small  $k$  values. Table 4 provides the thermal conductivities of various high explosives for comparison purposes.

**Table 4:** Thermal conductivities ( $k$ ) of various high explosives, RDX/HMX/TNT from *LASL Explosive Property Data*<sup>24</sup>.

Explosive	Thermal Conductivity, $k$ (W/m•K)
NTO	0.9298
RDX	0.1058
HMX	0.4180
TNT	0.2599

The critical temperature ( $T_C$ ) for symmetrical configurations of solid explosives may be determined using the Frank-Kamenetskii (F-K) formula<sup>11</sup> as seen in Equation 2 using the case for a short cylinder whose shape factor is 2.5. This method equates the heat losses from conduction to the heat generation from chemical decomposition giving the steady state solution.

$$T_C = \frac{Ea/R}{\ln \left[ \frac{r^2 * \rho * Q * A * Ea}{T_C^2 * k * Sh * R} \right]} \quad (2)$$

where  $T_C$  = Critical temperature [K]

$Ea$  = Activation energy [cal/mol]

$A$  = Preexponential factor [ $s^{-1}$ ]

$R$  = Gas constant [1.987 cal/K/mol]

$r$  = Radius or half thickness [cm]

$\rho$  = Density [g/cm<sup>3</sup>]

$Q$  = Heat of decomposition [cal/g]

$k$  = Thermal conductivity

$Sh$  = Shape factor [2.5]

An iterative solution is required for this formula since  $T_C$  appears on both sides of the equation. The values in Table 5 were used to estimate the critical temperature of the NTO pellets:

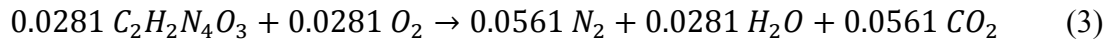
**Table 5:** Property values used to estimate the critical temperature of the NTO pellets, with some values taken from Smith and Cliff<sup>4</sup>, and Rohan<sup>6</sup>.

$T_C$ Estimation Parameters	
$Ea$	175 kJ/mol, <sup>4</sup>
$A$	$1.62 \times 10^{15} \text{ s}^{-1}$
$r$	0.9525 cm
$\rho$	1.79 g/cm <sup>3</sup> , <sup>6</sup>
$Q$	59.7 kJ/mol, <sup>4</sup>
$k$	0.9298 W/m•K = 0.0022315 cal/cm/sec/K

The reported critical temperature value for NTO is  $226.5 \pm 10.5^\circ\text{C}$ .<sup>4</sup> The critical temperature of the specific NTO pellets used for this research was estimated using Matlab and found to be  $211.5^\circ\text{C}$ . This approximation is  $15^\circ\text{C}$  less than the reported value, suggesting some uncertainty about what size charge the reported value was obtained. It should also be noted that due to variations in explosive charge size and the experimental testing conditions, some of the values used in the calculations (as seen in Table 5) were based on reported values that are noted to have some variability between research groups.

In terms of the amount of pressure generated by heating the pellets, it is generally understood that per 1 gram of explosive, 1 liter of gaseous products are produced. Therefore, to determine the amount of pressure generated by heating a single 3.65-gram NTO pellet inside the closed vessel (SB-IV), the following series of calculations was performed.

First, the complete reaction was assessed to determine the total moles of gaseous products. For this series of thermal cook-off testing, it is significant to note the effects of the free/extra volume (air) within the closed system is due to it being of greater size than the initial volume of the explosive pellet sample. It is also important to note that the reaction of the NTO is underoxidized, and can therefore supplement additional reactions within the free air: the presence of oxygen can react with excess carbon monoxide and free carbon from the underoxidized explosive. Equation 3 is the balanced chemical equation for NTO. Calculating the number of moles of the reaction products generated by heating a single pellet revealed there to be an inadequate amount of oxygen within the vessel. This prevents all of the product gases from completely burning, thus requiring that the heat evolved be calculated from the heat of reaction for the particular amount of oxygen available. The heat of combustion of NTO, specific to this reaction, is - 233.82kcal/mol. Using this value, Equation 4 (which incorporates water in liquid form as water is included in the reaction products) was used to solve for the total heat produced by the NTO pellet<sup>25,26,27</sup>:



$$Q' = Q - n_{H_2O} \lambda_{b,H_2O} = -n \Delta H_C^0 - n_{H_2O} \lambda_{b,H_2O} \quad (4)$$

where  $\Delta H_C^0$  = Heat of combustion

$Q'$  = Heat generated by reaction including  
latent heats.

$Q$  = Heat generated by reaction

$n$  = Moles of NTO

$n_{H_2O}$  = Moles of water

$\lambda_{b,H_2O}$  = Heat of vaporization of water

Solving for  $Q'$  to determine the amount of energy available to heat the gaseous products from reference state to adiabatic flame temperature, the total heat produced by a single NTO pellet was found to be 224.1 kcal.

Next, the temperature of the generated gases was used to determine the adiabatic flame temperature ( $T_a$ ) of the reaction by first computing the average heat capacity (Equation 5) of the reaction products as a function of temperature. Using this  $C_p$  relation to then formulate Equation 6, the cubic relation of Equation 7 could then be derived to determine the adiabatic flame temperature<sup>25,26</sup>:

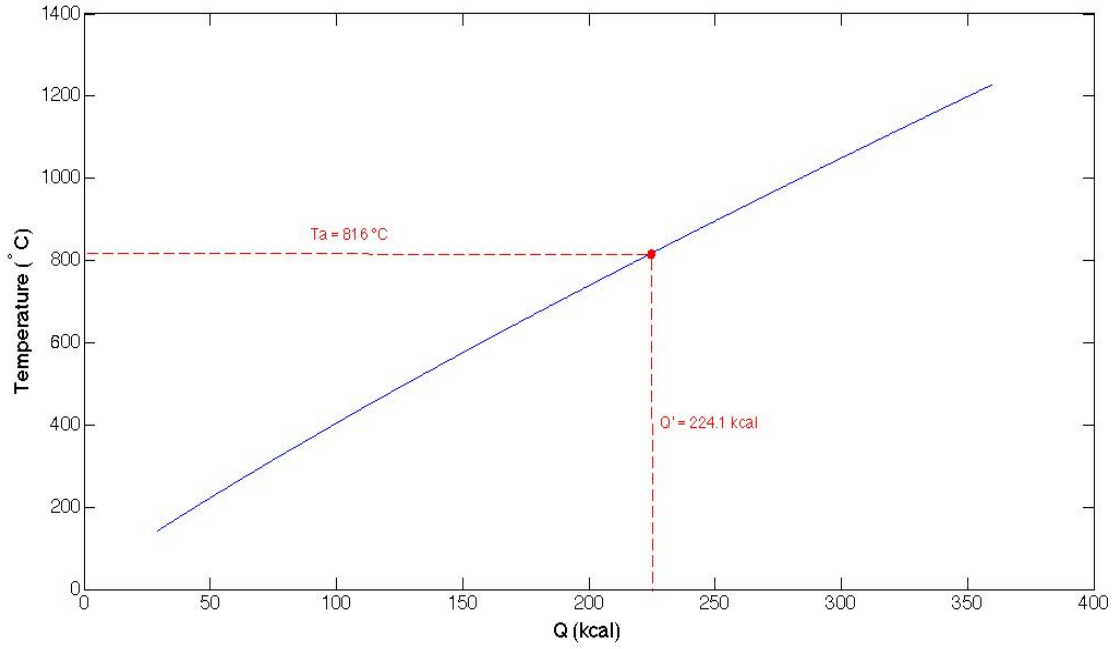
$$C_p = \sum_i n_i C_{p,i} \quad (5)$$

$$Q' = n \int_{T_0}^{T_a} C_p dT \quad (6)$$

$$Q' = n \left( a(T_a - T_0) + \frac{b}{2} (T_a^2 - T_0^2) + \frac{c}{3} (T_a^3 - T_0^3) \right) \quad (7)$$

The  $a$ ,  $b$ , and  $c$  variables of Equation 7 are empirical constants for molar heat capacities of gases at constant pressure<sup>25</sup> where  $T$  is in degrees Kelvin obtained from Equation 5. As such, the computed adiabatic flame temperature was found to be 816°C via the plot in Figure 6.





**Figure 6:** Adiabatic flame temperature approximation for NTO pellets using calculated heat generated ( $Q'$ ).

Now, to correct the above calculations for constant volume, the ratio of specific heats ( $\gamma$ ) of the detonation product gases was determined using the following relation<sup>25</sup>:

$$\gamma = \sum_i n_i \gamma_i \quad (8)$$

From  $\gamma = 1.3483$ , the temperature of the gas in the final pressurized state is found by multiplying  $\gamma$  by  $T_a$  yielding  $T_v$ , the adiabatic flame temperature at constant volume, 1195.3°C. This value is then used to ultimately determine the generated internal vessel pressure using the Nobel-Able Equation of State (EOS, Equation 9)<sup>25</sup>:

$$P(V - \alpha w) = nRT \quad (9)$$

where  $\alpha$  is the covolume of gas, and  $w$  the weight of the gas. Final results indicate that the generated pressure of a slowly heated, single NTO pellet (deflagrating) is about 400

psi. However, it is important to note that for these pellets, were they to detonate rather than deflagrate, they would generate a much larger pressure.

Lastly, to estimate the time to heat a single pellet to an internal subcritical, uniform temperature of 160°C, the following relation was applied, assuming heating via conduction as stated previously<sup>23</sup>:

$$\Delta t = \frac{mC_p\Delta T}{Q_{cond}} \quad (10)$$

By applying the previously calculated  $\dot{Q}_{cond}$  value, change in temperature ( $\sim 140^\circ\text{C}$ ), specific heat ( $C_p$ ), and pellet mass (3.65 g), the total time ( $\Delta t$ ) to heat the solid pellet is about 102 s, just less than 2 min.

Upon achieving this uniform temperature is when the cook-off process begins by increasing the heating rate at a specific value (e.g. 3°C/hr, 4°C/hr, etc.) until the critical temperature is achieved—a point at which the pellet will either deflagrate or detonate. As such, the total duration of a single test for slow cook-off can range anywhere from 12 to 20 hours. For fast cook-off tests, total test time is greatly reduced to as few as a couple of hours.

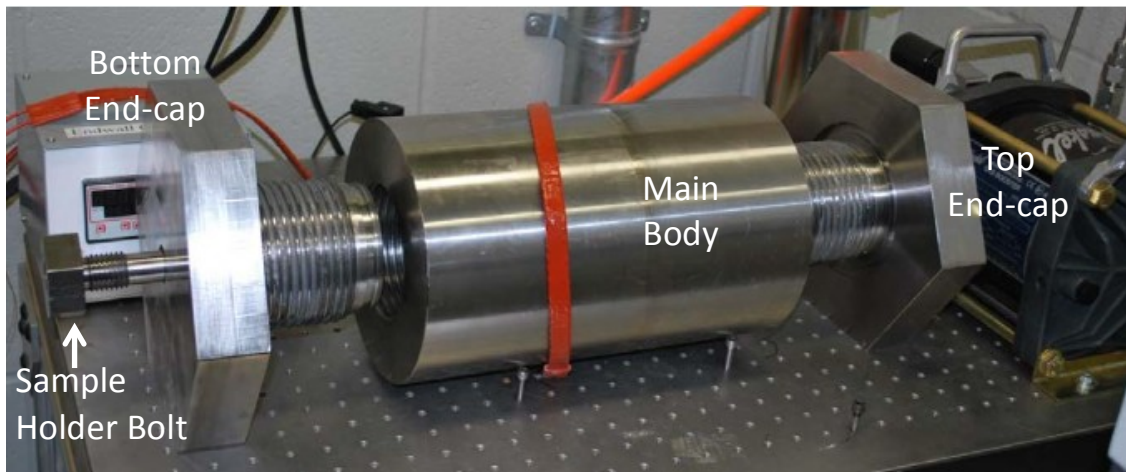
### Testing Vessel

The test vessel in which cook-off tests were performed for this study was the Petersen Research Group Strand Burner IV (SB-IV) at Texas A&M University. Strand burners are ideal, constant-volume pressure vessels used for characterizing the behaviors of propellants.<sup>21</sup> However, the decision to use the strand burner for cook-off testing was for safety concerns regarding the possibility of a pellet detonating rather than

deflagrating, as well as the need to heat the samples to extreme temperatures. SB-IV is a high-pressure strand burner capable of testing at pressures up to 10,000 psi with a maximum allowable stress that incorporated a safety factor of four.<sup>21</sup> The vessel consists of 17-4 PH stainless steel due to its significant yield and tensile strengths. A complete list of SB-IV ratings may be found in the thesis by Dillier.<sup>21</sup>

The SB-IV consists of a main body with an internal diameter of 3.7 in and outer diameter of 7.5 in, as well as two internally threaded end-caps of the same 8.5 in diameter by 1.75 in thick hexagonal head; the overall length of the strand burner is 15.5 in. The burner is equipped with high-strength polyurethane O-ring seals that are resistant to wearing and tearing, and have a maximum exposure temperature of about 90°C. While the temperatures achieved during cook-off are well above 200°C, these O-rings are not in direct access to the internally heated air nor the cartridge heater generating the heat.

The end-caps of the strand burner, while of the same overall dimensions, have different ports. The top end-cap has two ports: one which is used for feeding through the wires of the cartridge heater, and the other (divided via a T-fitting) for venting the vessel of generated product gases and pressure, as well as for the purpose of inserting a type K thermocouple. The location of this specific thermocouple monitors the temperature of the air within the SB-IV. The bottom end-cap has a 1-1/16 in-12 UNF centrally tapped hole in which the sample holder is inserted. Figure 7 shows the expanded view of SB-IV's aforementioned main components.



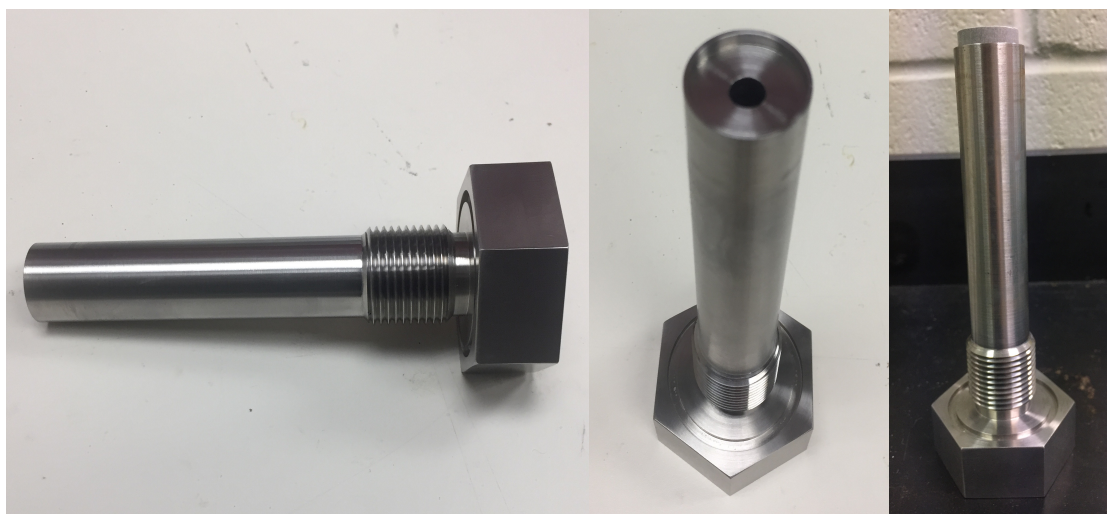
**Figure 7:** SB-IV expanded view taken directly from Dillier.<sup>21</sup>

When assembled and used for testing, SB-IV is attached to a vertical stand using aluminum braces equipped with L-brackets. The entire SB-IV apparatus weighs approximately 220 lbs. Since the main purpose of the strand burner is to burn propellant strands, the internal surface of the main body and end-cap faces are coated in nickel plating so as to prevent corrosion from combustion product residues; this coating is also beneficial for cook-off testing as the heated pellets, which will either deflagrate or detonate, will also generate corrosive products.

### **Sample Holder**

The sample holder, that is screwed into the base end-cap of the SB-IV, is a 2-in hex head bolt custom-machined from a 2.5-in diameter, 17-4 PH stainless steel rod. The threads of the sample holder are 1-16 in-12 UNC threads that are wrapped with Teflon tape for sealing purposes. While the testing vessel is not initially pressurized for cook-off tests, the addition of a 1.5-in diameter O-ring groove was machined into the sample

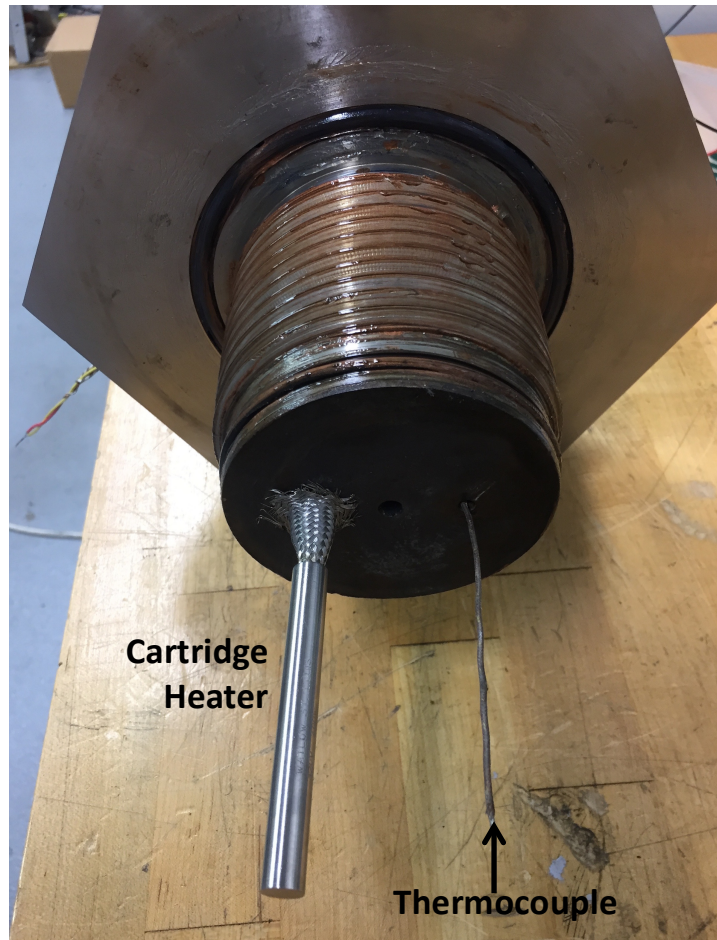
holder to aid in sealing from the pressure generated by the heated pellet. At the tip of the sample holder bolt is a bored hole, 0.8-in diameter and 0.1-in deep, that holds a 0.75-in dia. by 0.375-in thick NTO pellet. Through the entire length of the bolt runs a central hole in which a thermocouple probe is inserted; this type K thermocouple is in direct contact with the pellet to continuously monitor its temperature for the duration of the cook-off test, ultimately detecting the sudden spike in temperature revealing the sample's critical temperature. Figure 8 depicts the custom-machined sample holder used specifically for cook-off tests herein, with the schematic available in Appendix A.



**Figure 8:** Sample holder bolt. Far right image depicts actual configuration of bolt holding NTO pellet sample.

### **Cartridge Heater and Temperature Controller**

To heat the pellet samples, a cartridge heater was internally suspended from the top end-cap of the SB-IV, as seen in Figure 9, next to a separate type K thermocouple that was used to measure the air temperature within the vessel.

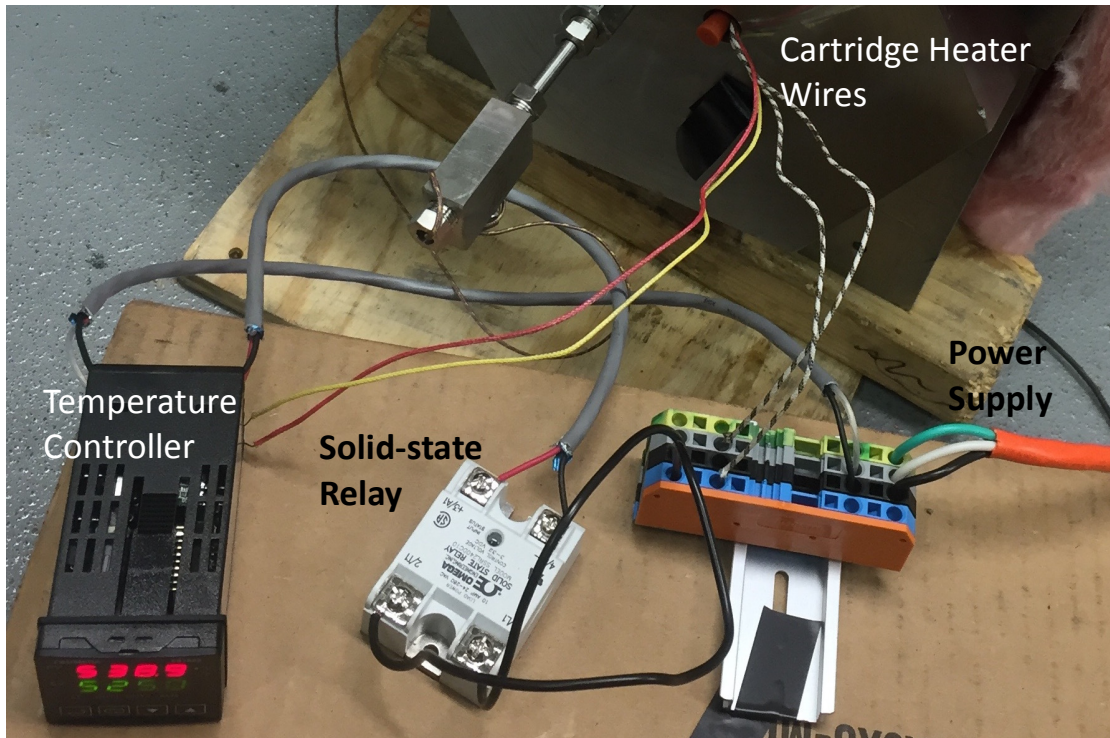


**Figure 9:** Internal cartridge heater and air-temperature-measuring thermocouple locations on top end-cap of SB-IV.

A Watlow Firerod cartridge heater—400 watts, 120 volts AC, 3/8-in diameter by 3-in length—with an internal type K thermocouple sensor was paired with a Omega Engineering CN7823 Ramp/Soak Controller to monitor and control the temperature output by the heater. A solid-state relay was wired between the controller and the heater to make the required AC/DC power change. The controller (with the cartridge heater connected) was then auto-tuned in proportional-integral-derivative (PID) mode so as to achieve the ideal response from the control system to prevent the cartridge heater from



overshooting the setpoint temperature when initially heating. The controller was then used to increase the temperature of the cartridge heater by the specified heating rate, e.g. 3°C/hr, depending upon the type of test being performed, i.e. fast or slow cook-off. The heater, controller, and solid-state relay setup may be seen in Figure 10.



**Figure 10:** Electrical configuration for temperature controller and cartridge heater.

### **Data Acquisition Tool**

The data-recording tool utilized for the cook-off experiments was the Omega HH378 4-channel Temperature Data Logger complete with USB interface with Windows Software. This data logger, capable of recording data at a rate of 1 sample per second for the entire duration of each test, collected data from two separate thermocouples. The first thermocouple being the one which monitored the air

temperature inside the SB-IV; the second being the thermocouple fed up through the sample holder bolt, monitoring the temperature at the base of the NTO pellet. When testing, the data logger is connected to a computer with the appropriate software installed where it continuously monitors the temperature of both thermocouples, generating an active, live-feed graph. These graphs indicate the point at which the pellet has reached its critical temperature by revealing a sudden spike in temperature from both thermocouples; this spike is due to the sudden increase in temperature via the heat generated by the NTO pellet either deflagrating or detonating. However, due to lack of visibility within the vessel during testing and lack of fast-response instrumentation, the only way to determine if a pellet has detonated rather than deflagrated is by listening for a loud, audible “pop.”

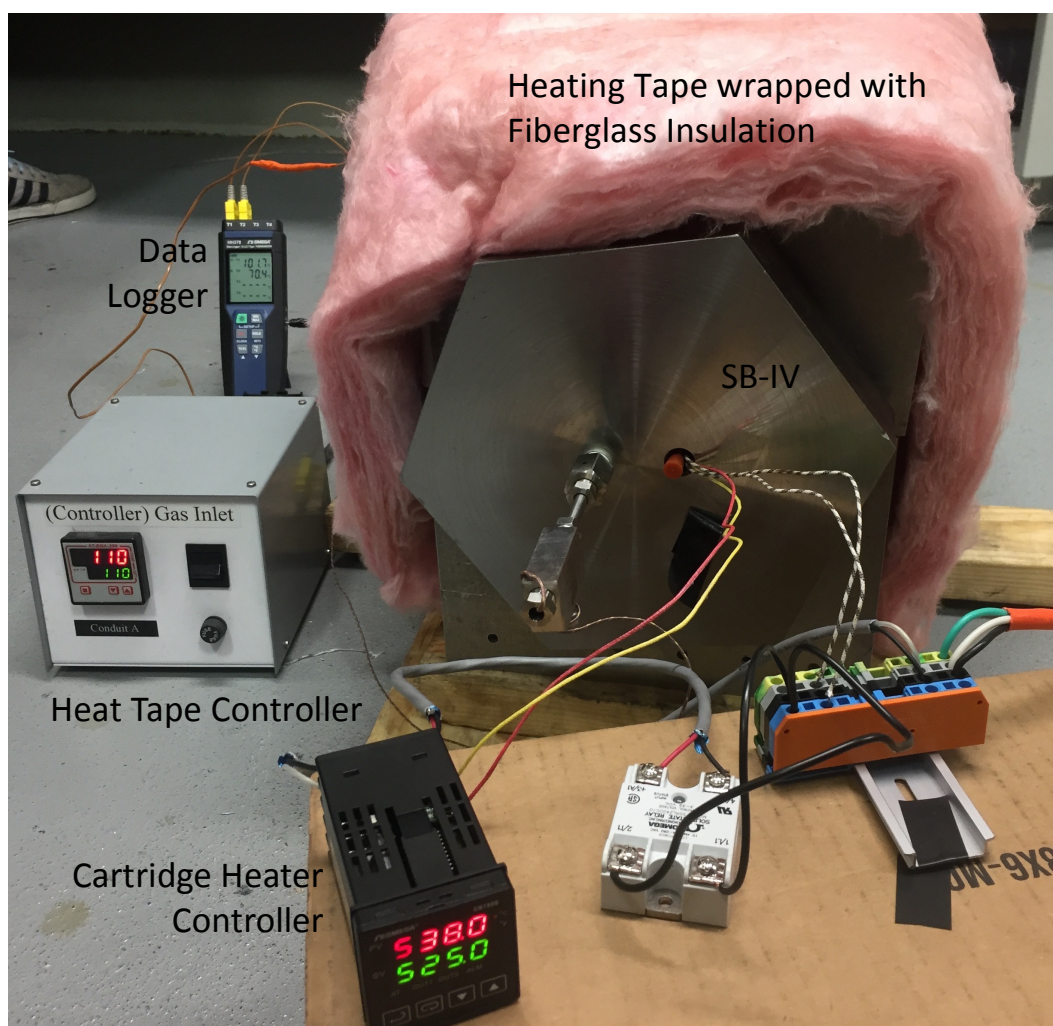
### **Experiment Optimization**

While the main focus of this research was to study the cook-off effects of the pressed NTO pellets to provide greater insight for CHEC applications, a significant amount of time was spent preparing for the general tests. The need to heat the internal chamber of the SB-IV to temperatures greater than 200°C proved to be rather challenging as much of the heat generated by the cartridge heater was absorbed by the thick walls of the strand burner. A variety of solutions were tested to combat this problem, including the application of an external heat source and internal insulation.

The first of these solutions tested was the application of an external heat source in addition to the existing internal cartridge heater. High-temperature heating tape was

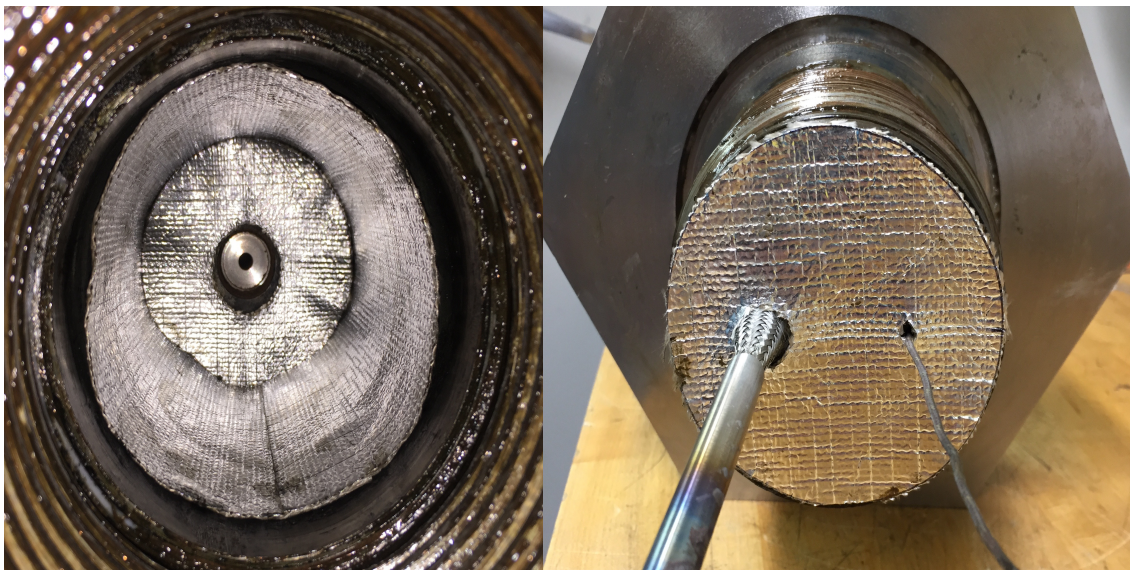


wrapped around the exterior of the main body of the SB-IV, which was further wrapped with off-the-shelf insulating material. The heating tape was set to a temperature of 110°C and was monitored and powered using a separate electronic controller. The vessel was allowed to warm for a period of six hours before powering on the cartridge heater. Unfortunately, while there was a sizeable increase in the temperature of the air within the SB-IV, the system was unable to achieve the required temperature. This system setup with the external heat supply may be seen in Figure 11.



**Figure 11:** External heat source system setup.

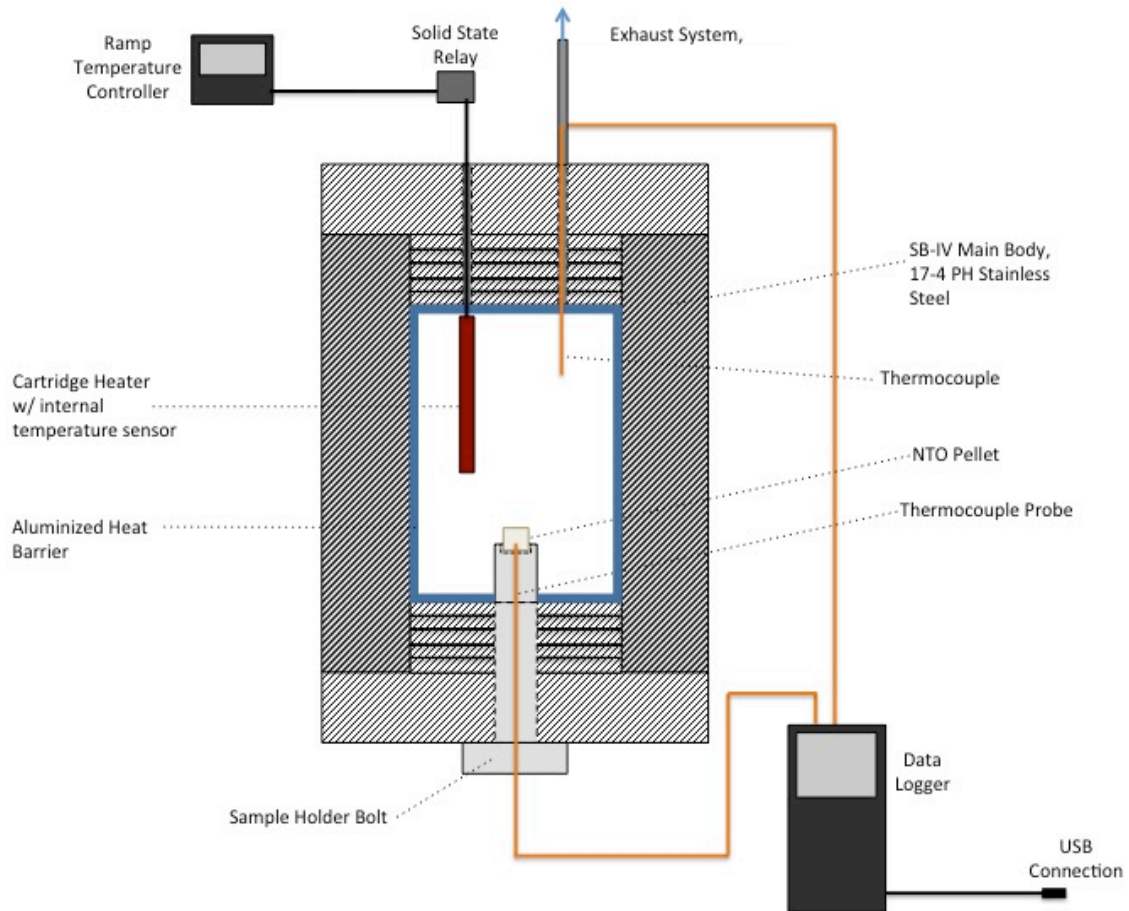
Another solution involved insulating the strand burner from the inside. A variety of materials were researched for this application including ceramic fiber insulators, high-temperature silica insulation, and an aluminized heat barrier. The aluminized heat barrier was found to be the most effective in this case, achieving internal air temperatures greater than 200°C. This improvement was due to the material having a highly reflective surface used to reflect radiant heat and withstand radiant temperatures up to 2000°F (1093°C). As such, this heat barrier was used during cook-off tests to line the inner walls and end-cap faces of the SB-IV, as seen in Figure 12.



**Figure 12:** Aluminized heat barrier lining inside of SB-IV. Left image looks down into strand burner; right image depicts internal face of top end-cap.

Prior to conducting the actual cook-off tests of NTO, a series of preliminary tests was performed using a glass quartz pellet—in place of an NTO pellet—to safely record the temperature changes during the specified temperature rate without having to work

with a live NTO pellet. The complete testing schematic (Test Schematic 1) may be seen in Figure13.

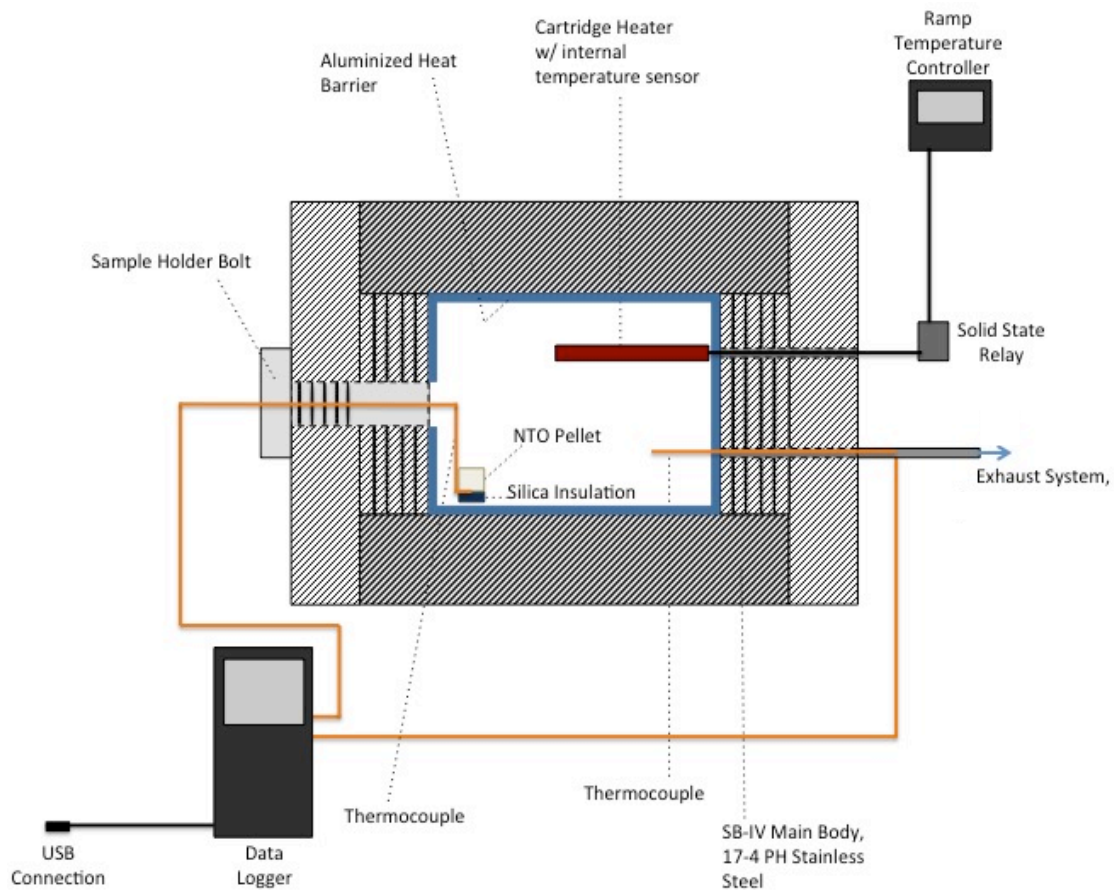


**Figure 13:** Test Schematic 1: vertically oriented SB-IV with pellet situated on sample holder bolt.

These mock tests ensured that all components were functioning properly, that the required temperatures within the vessel were high enough to ensure successful cook-off of the NTO pellets, and that the heating rate used to increase the temperature of the cartridge heater was similarly reflected in the temperature of the pellet. However, after a period of testing, the glass pellet, when mounted on the bolt/holder, was unable to



achieve the required temperatures to initiate cook-off. The glass pellet was therefore moved further into the strand burner along with the thermocouple—while still fed through the central port on the sample holder bolt—simply taped to the underside of the pellet, and backed by a small square of high temperature silica insulation material to ensure the thermocouple was detecting the temperature of the pellet. For the first setup (Figure 13), it is believed that much of the heat was absorbed by the small portion of the sample bolt that was exposed to the heated air, thereby preventing the pellet from heating effectively. For the second setup (Figure 14), the strand burner was reoriented to operate on its side, in horizontal form.



**Figure 14:** Test Schematic 2: horizontally oriented SB-IV with resting inside vessel.

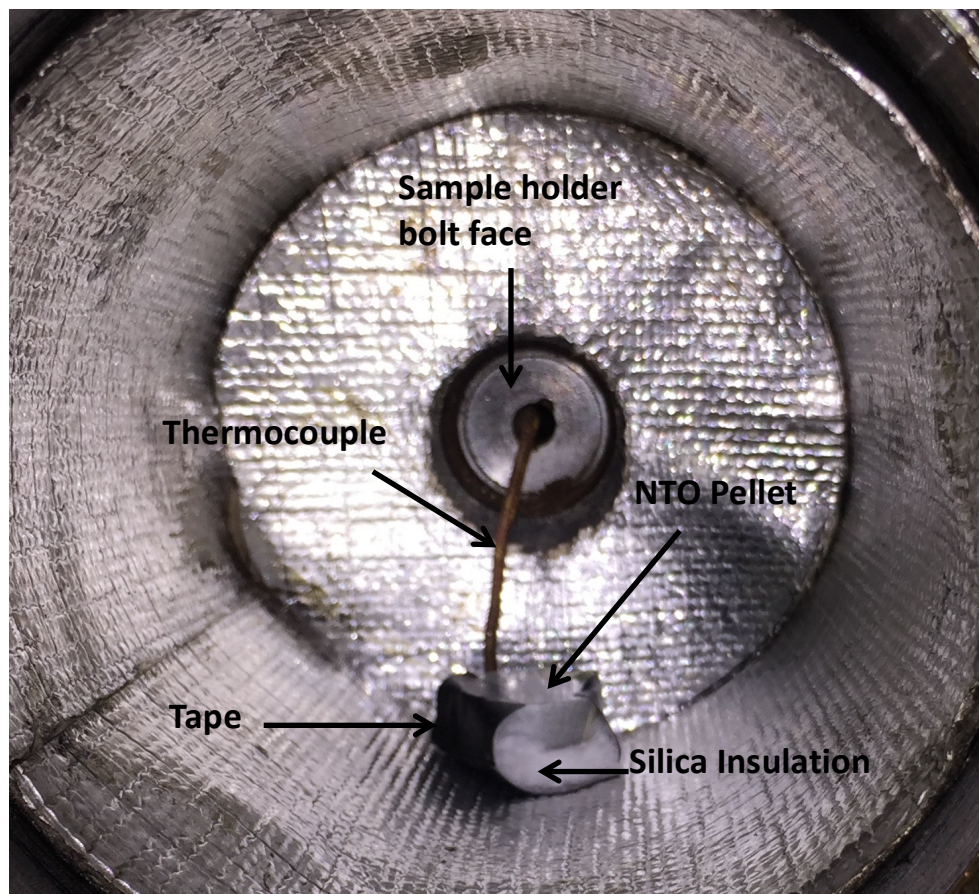
## CHAPTER IV

### EXPERIMENTAL TESTING

#### **Cook-off Testing Procedures**

All tests were performed within the SB-IV, located inside a blast-shielded test chamber. All cook-off operations were remotely controlled from an adjacent control room for safety purposes. As noted in Chapter II of this thesis, the reported critical temperature of NTO is  $226.5 \pm 10.5^{\circ}\text{C}$ <sup>16,4</sup>; however, after performing the first two cook-off tests in this research, the NTO pellets were found to ignite/detonate at significantly lower temperatures, one as low as  $175^{\circ}\text{C}$ . While the exact reasoning for such a phenomenon is unknown, it is reasonable to assume that, as with many other types of explosives, performance parameters can oftentimes be affected by the size and shape of the sample tested. As such, the predetermined plan to start heating the pellet from the steady state temperature of  $210^{\circ}\text{C}$  was altered to account for such an unexpected temperature difference. Therefore, the following section outlines the method of testing redeveloped specifically for these NTO pellets for this specific cook-off test setup.

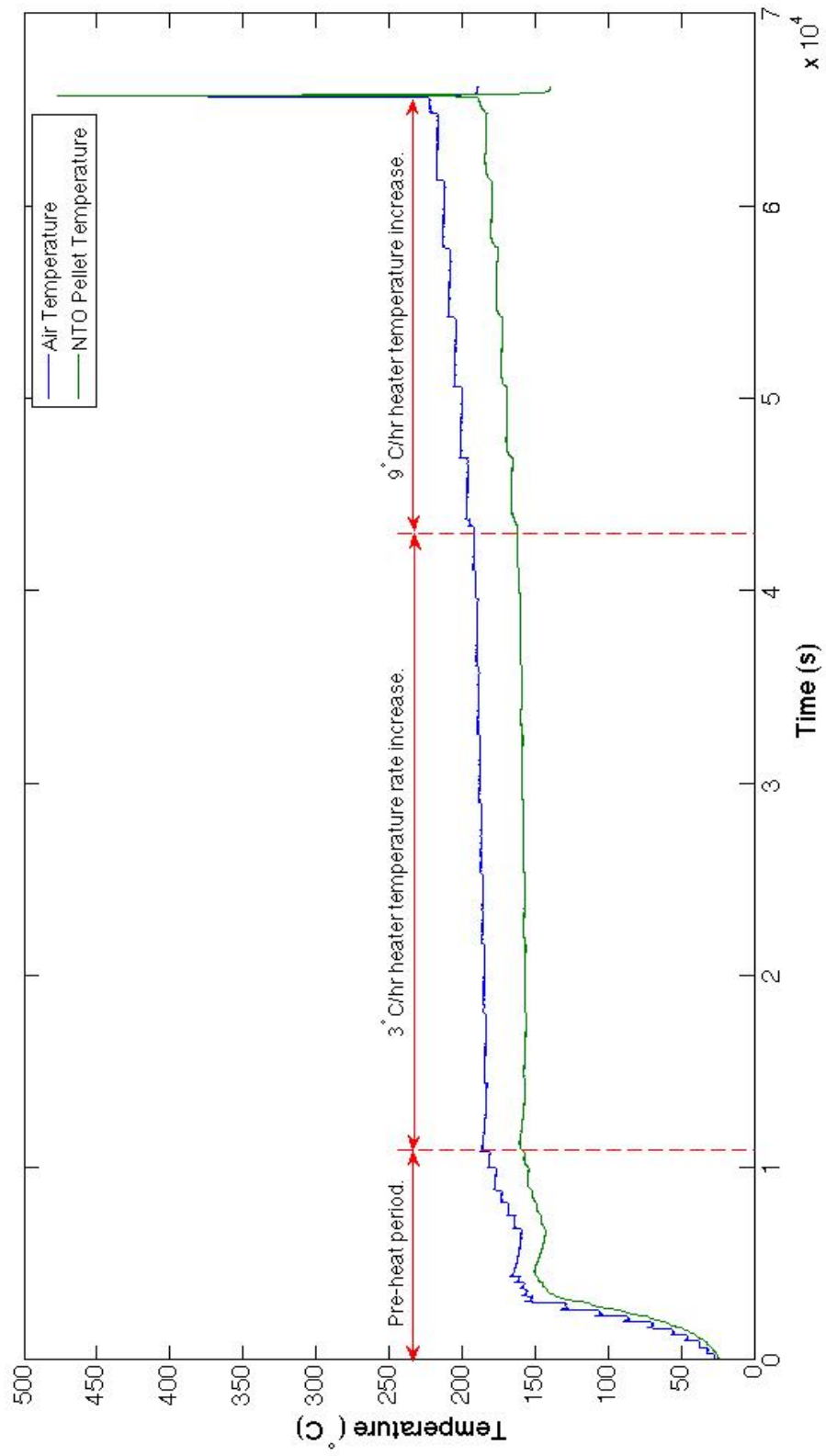
To begin testing, the sample NTO pellet was prepared by securely attaching the thermocouple to the bottom surface of the pellet, which was then inserted into the vertical SB-IV. Figure 15 depicts the view of the inside of the SB-IV of how the NTO pellet was oriented for testing.



**Figure 15:** Second setup with NTO pellet resting inside main chamber.

With the pellet secured inside the chamber, the data logger was set to begin recording temperature data from both thermocouples as the cartridge heater was gradually heated to a steady temperature, that translated to a subcritical, uniform pellet temperature of 160°C. This period is termed the preheat period. The starting temperature of 160°C was chosen based upon an initial test where the NTO was found to decompose at 175°C, as mentioned above; therefore, 160°C provided a temperature range of  $\pm 15^\circ\text{C}$  around which the pellet was incrementally heated to determine the critical temperature. Due to the fact that the cartridge heater's temperature sensor was located internally

within the device, the temperature to which the cartridge heater was set was significantly higher than the actual temperature of the heat being radiated from the heater. As such, one of the samples tested was used to determine the heating rate at which the pellets exhibited the best isothermal heating. As seen in Figure 16, the pellet underwent preheating, followed by the specified 3°C/hr temperature increase in the cartridge heater. This specific heating rate found that for every 3°C/hr the cartridge heater temperature was increased, the pellet only exhibited a 1°C/hr increase in temperature. At this rate, the duration of a single test was drastically extended. Therefore, to ensure the 3°C/hr increase within the pellet sample itself, the cartridge heater temperature was increased at a rate of 9°C/hr until cook-off was achieved. Thus, all following cook-off tests were initiated using the initial preheat period, immediately followed by the 9°C/hr temperature increase of the cartridge heater.



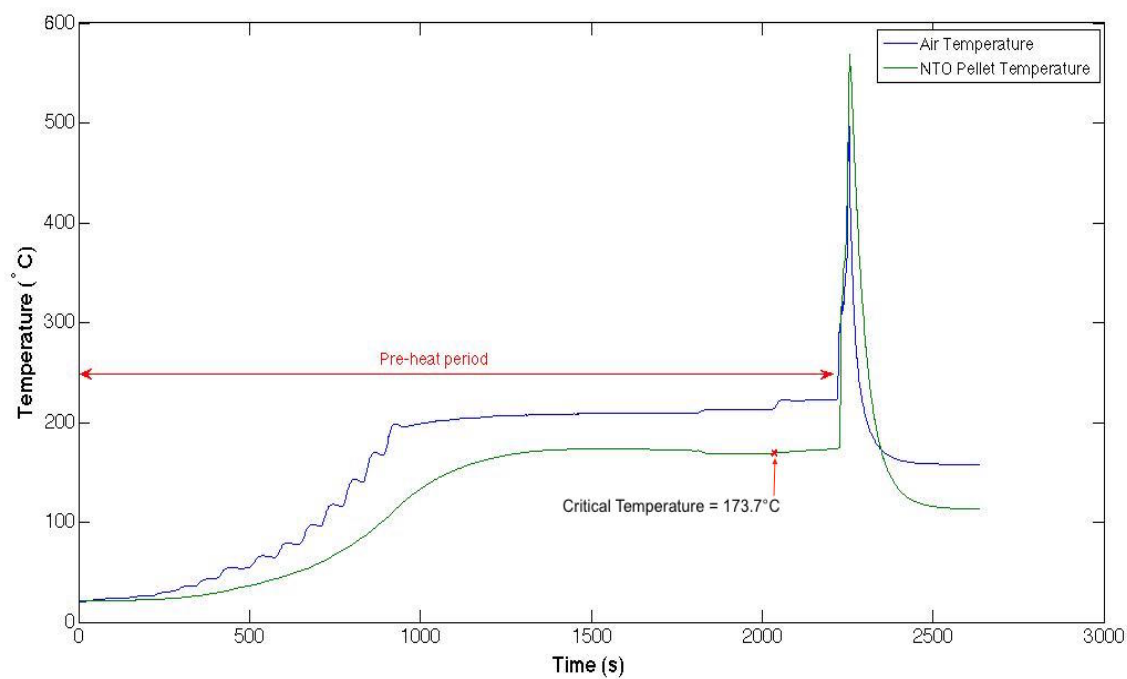
**Figure 16:** Preliminary cook-off test used to determine appropriate heating rate for NTO pellet.



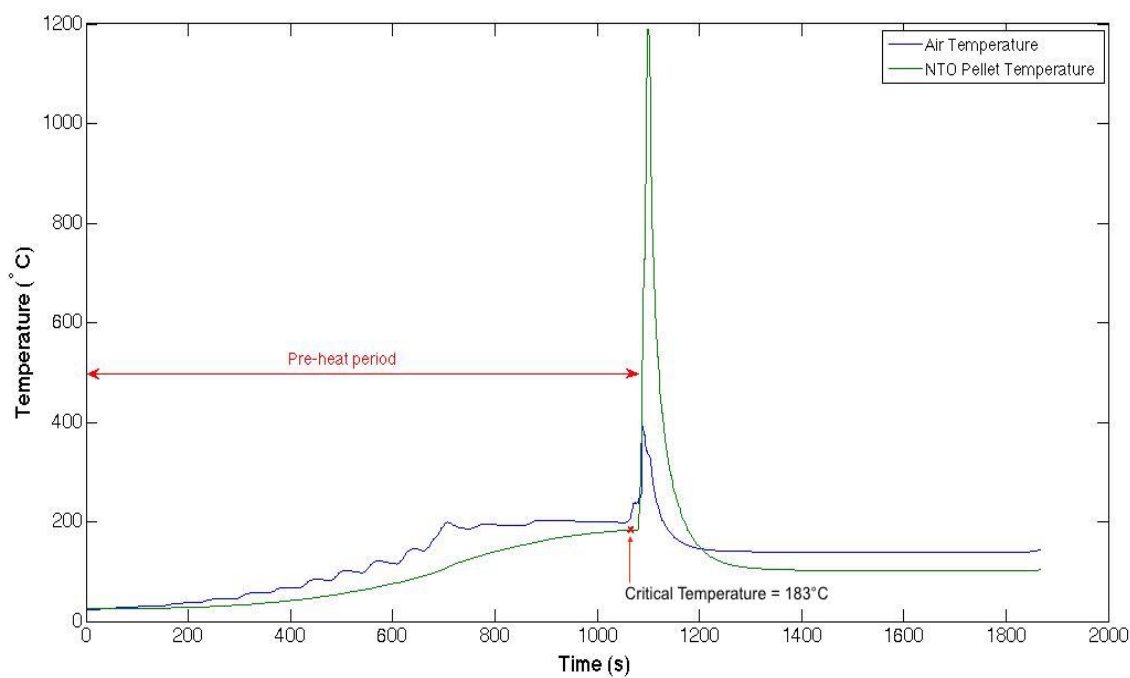
It is significant to note that the heating rate used in testing remained constant throughout the entire duration of each specific test. The temperature, as it increases with time, eventually reaches the point at which the sample responds by either deflagrating or detonating, indicating the critical temperature value of that specific sample. Depending upon the heating rate utilized, each individual test can range anywhere from a matter of a few hours for fast cook-off to more than twelve hours for slow cook-off. Regardless, the point at which the samples reach their critical temperature is indicated by an incredibly large, sudden increase in both the pellet and air temperatures detected within the vessel.

### **Test Results and Data Analysis**

Prior to conducting these cook-off tests, the full-scale behavior of the NTO pellets was not entirely known. It was expected that the pellets would either deflagrate or detonate and have the performance parameters of those reported in literature and estimated in Chapter III, but the scale of their severity was only completely understood once actually tested. The plots in Figures 17 and 18 indicate the first two NTO cook-off tests wherein both samples achieved their critical temperature during the rapid preheat phase. This premature light-off was due to the preheat of the sample occurring too quickly at the rate of 50°C/min; at this rate, the top surface of the pellet was heated, not allowing adequate time for the heat to transfer through the entire thickness of the pellet, causing the top pellet surface to initiate cook-off, in turn triggering the entire reaction.

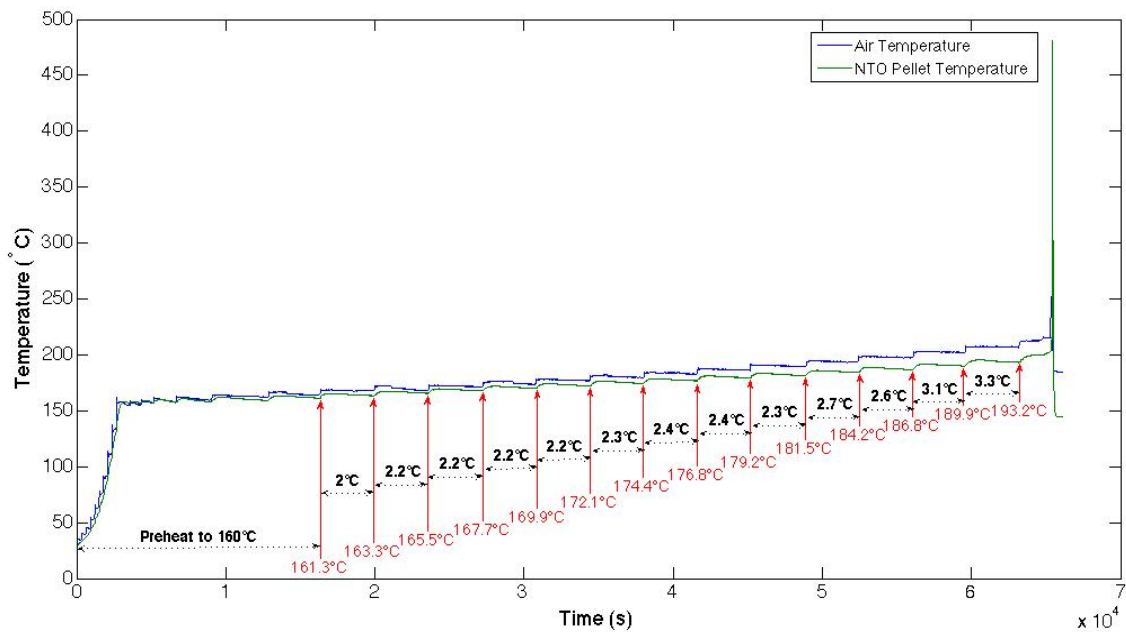


**Figure 17:** Test 1—NTO pellet undergoing cook-off during preheat period.

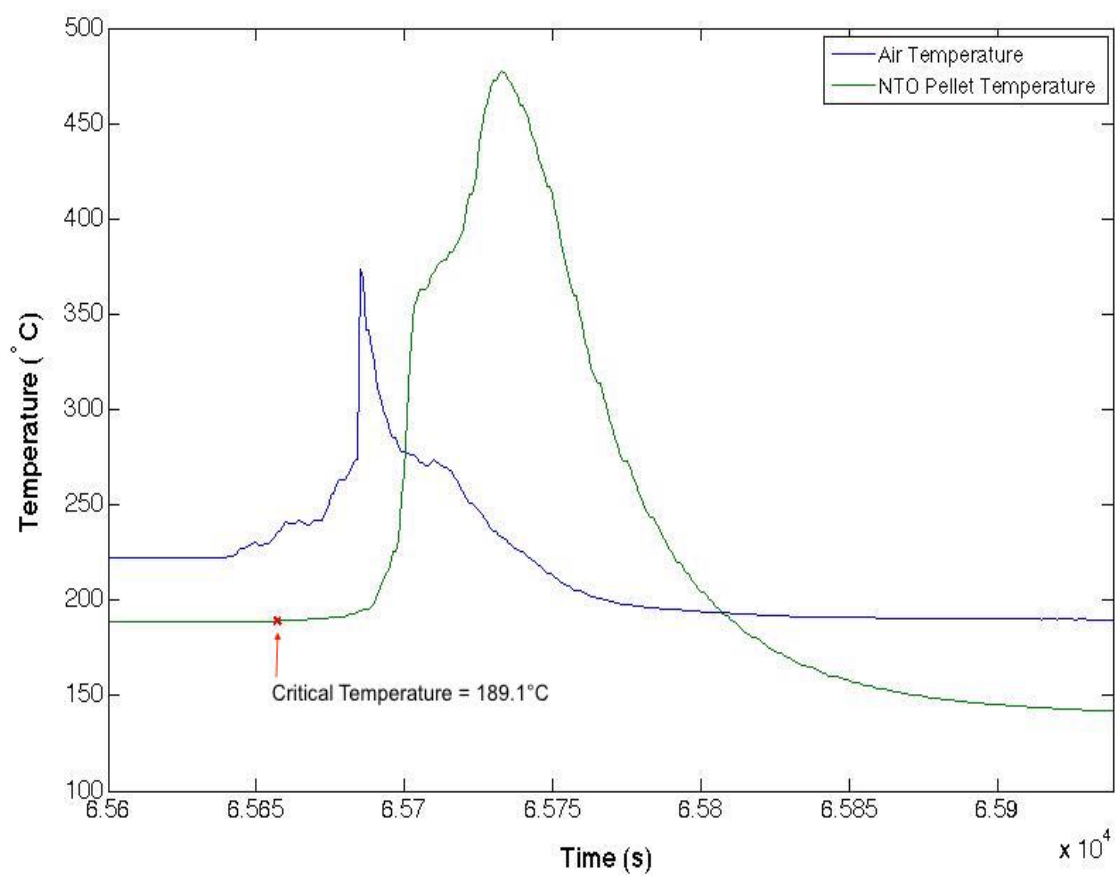


**Figure 18:** Test 2—NTO pellet undergoing cook-off during preheat period.

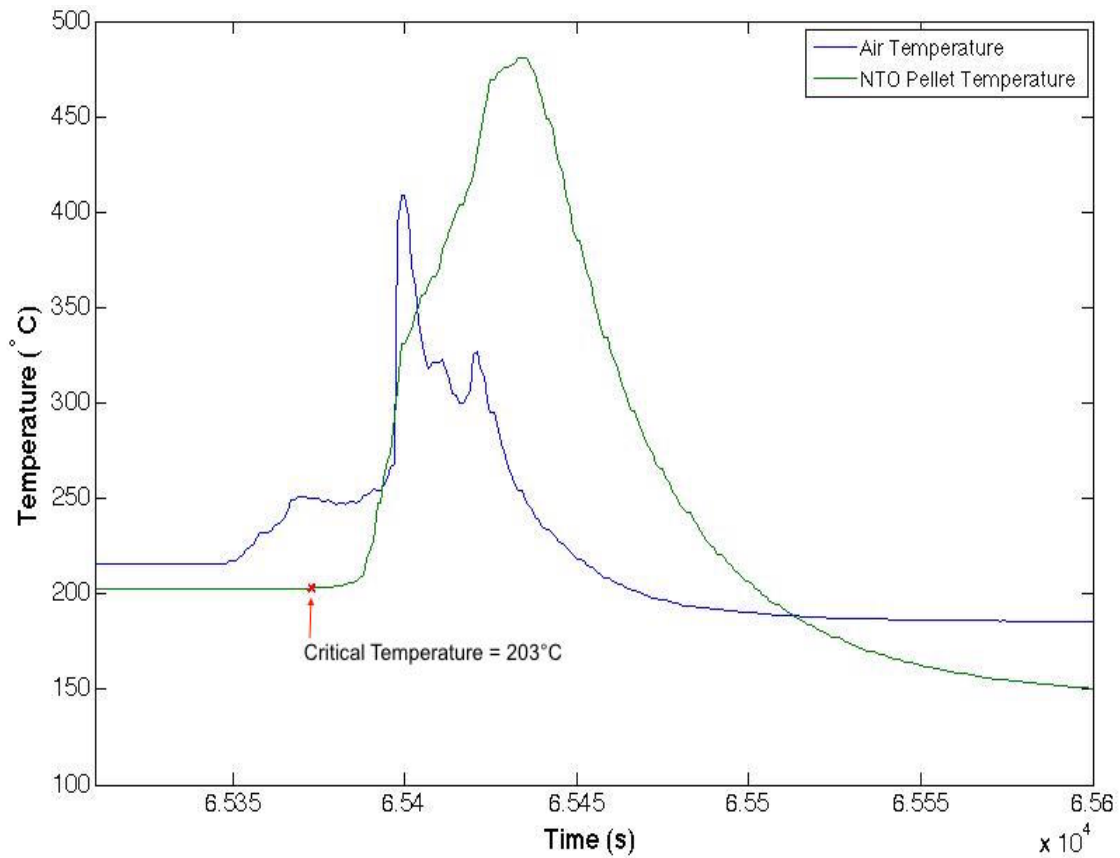
Based upon the information obtained from Tests 1 and 2, the preheating rate employed for Tests 3 and 4 was significantly slowed to a rate of 50°C/5min. This rate allowed the pellet to steadily heat up to the uniform temperature of 160°C prior to starting the incremented heating rate of 9°C/hr. Figure 19 provides a complete look at the temperature increase experienced by the pellet every hour the cartridge heater was increase at this rate of 9°C/hr. Figures 20 and 21 present the data obtained for slow cook-off Tests 3 and 4, where heating was initiated at a rate of 9°C/hr (directly correlating to a 3°C/hr increase in pellet temperature). For all four slow cook-off tests, all pellets were found to have deflagrated.



**Figure 19:** Complete slow cook-off temperature increase in NTO pellet per hour at cartridge heater temperature increase of 9°C/hr.

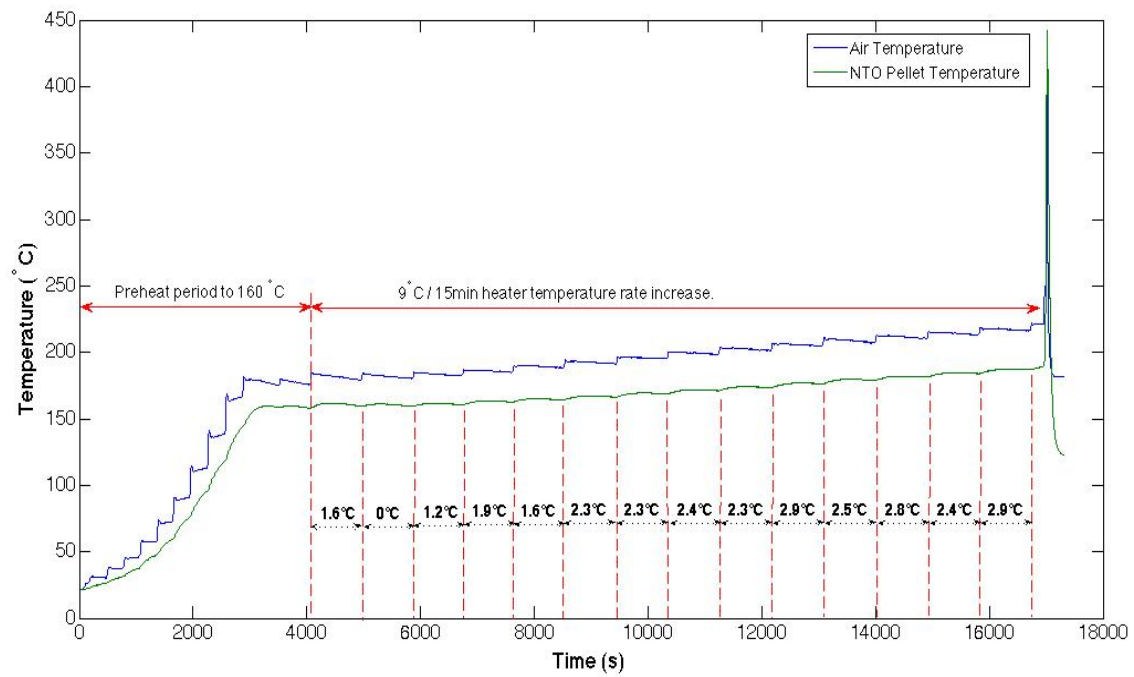


**Figure 20:** Test 3—NTO pellet undergoing slow cook-off at rate of 9°C/hr.

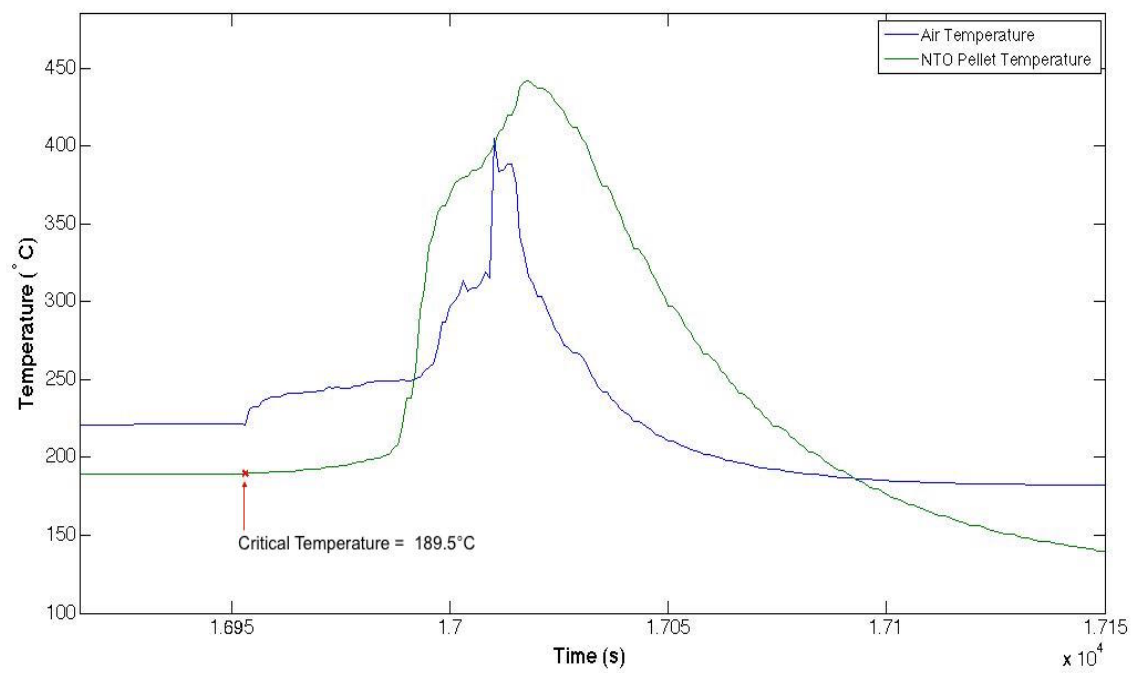


**Figure 21:** Test 4—NTO pellet undergoing slow cook-off at rate of 9°C/hr.

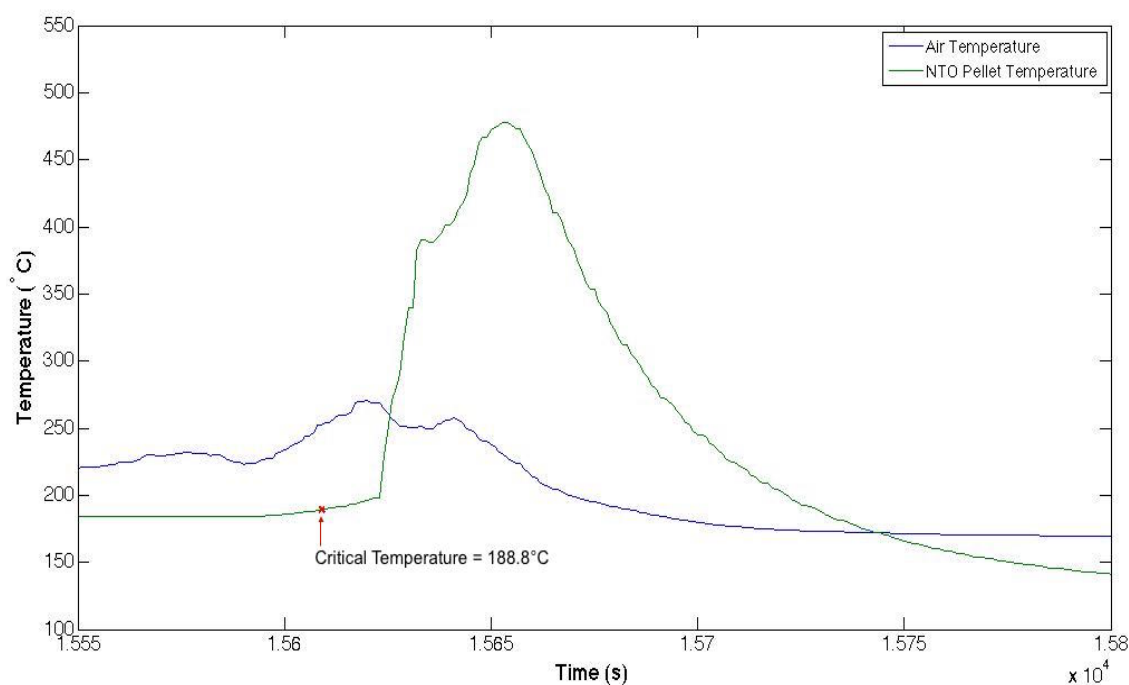
Two fast cook-off tests were performed at a rate of 9°C/15min, using the same preheat method as slow cook-off where the NTO pellet was heated to a uniform temperature of 160°C. Figure 22 reveals the exact temperature increase experienced by the pellet with every 9°C increase in cartridge heater temperature every 15min. Figures 23 and 24 reveal the critical temperatures obtained from Tests 5 and 6.



**Figure 22:** Complete fast cook-off temperature increase in NTO pellet per 15min at cartridge heater temperature increase of 9°C/15min.



**Figure 23:** Test 5—NTO pellet undergoing fast cook-off at rate of 9°C/15min.



**Figure 24:** Test 6—NTO pellet undergoing fast cook-off at rate of 9°C/15min.

Figure 25 shows an inside look of how the SB-IV and aluminized heat barrier held up with each cook-off test. As seen in the image, the reflective surface of the barrier was destroyed by the extreme heat generated by the decomposition of the NTO pellet, and thus required replacing with each test. Figure 26 is evidence of the deflagration of the pellets. When an explosive undergoes a complete decomposition reaction, there should be minimal to no residue remaining; however, in this study, the stiff, foam-like substance remaining after each test indicates that the pellets may possibly be experiencing premature decomposition resulting in an incomplete reaction. Premature decomposition may be attributed to a variety of factors including localized, uneven heating creating hot spots within the pellet, pressure buildup within the SB-IV created by gases emitted from the heated pellet, or the effect of confinement created by the SB-IV.

Another possible cause of premature chemical reaction may be due to the fact that the NTO pellets contain 0.5% calcium stearate and 0.5% graphite; calcium stearate has a significantly lower melting point (180°C) than NTO or graphite, suggesting that the calcium stearate may possibly be transforming into a molten form accelerating the pellet's rate of decomposition. Further testing could potentially indicate the cause of early decomposition.



**Figure 25:** Cook-off test damage to reflective surface of aluminized heat barrier.



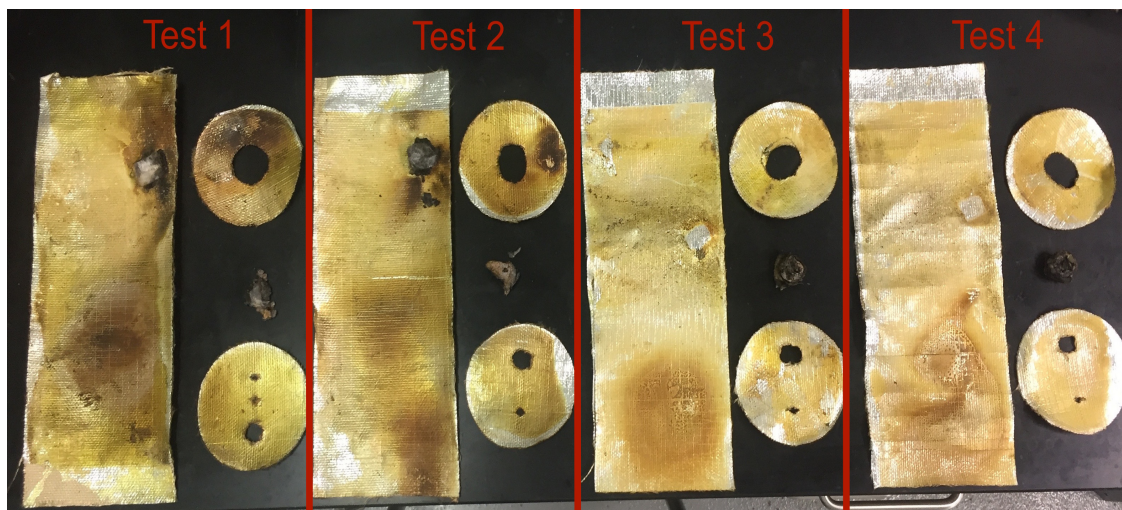


**Figure 26:** Cook-off reaction byproducts of NTO pellets.

Table 6 provides a collection of the critical temperatures recorded from the 4 slow cook-off tests, followed by Figure 27 displaying the complete damage caused by each test to the aluminized heat barriers.

**Table 6:** Slow cook-off Tests 1-4 critical temperatures and test duration.

Test #	Cartridge Heater Rate of Temperature Increase	Respective Pellet Rate of Temperature Increase	Critical Temperature ( $T_C$ , °C)	Time to Deflagration
1	Preheat phase	N/A	173.7	2,224 sec = 37.1 min
2	Preheat phase	N/A	183	1,079 sec = 18 min
3	9°C/hr	2-3°C/hr	189.1	65,657 sec = 18.2 hr
4	9°C/hr	2-3°C/hr	203	65,373 sec = 18.2 hr



**Figure 27:** Tests 1-4 cook-off damage.

Table 7 provides a collection of the critical temperatures recorded from the fast cook-off tests.

**Table 7:** Fast cook-off Tests 5 & 6 critical temperatures and test duration.

Test #	Cartridge Heater Rate of Temperature Increase	Respective Pellet Rate of Temperature Increase	Critical Temperature ( $T_c$ , °C)	Time to Deflagration
5	9°C/15min	1-3°C/15min	189.5	16,953 sec = 4.7 hr
6	9°C/15min	1-3°C/15min	188.8	15,608 sec = 4.3 hr

### Uncertainty

As with all experiments, there is inherent uncertainty when taking measurements, especially involving high explosives. The uncertainty in high explosives stems from the variation in performance parameters for a specific material which is dependent upon the size and shape of the charge. Additionally, the composition of the HE alone can generate uncertainty due to the possible presence of impurities; impurities may become

introduced during EM synthesis, or even during the pressing phase in the case of these pressed pellets. While the critical temperature of only a few NTO pellets was determined at the time of this thesis, the ability to minimize any variations in data for future testing would require testing a minimum of 20 pellets at a specific heating rate, ensuring to record any variations in mass or dimension. Based on the tests obtained via slow cook-off, the average critical temperature was determined to be  $196 \pm 7^\circ\text{C}$ ; fast cook-off revealed the average  $T_C$  to be  $189 \pm 1^\circ\text{C}$ .

Additional uncertainty exists in the early stages of heating the NTO pellets. Based upon the explosive residue remaining after each test and the low critical temperatures recorded in comparison to those reported in literature, it is determined that the NTO pellets are experiencing premature decomposition. Specifically for the slow cook-off tests, this may be a result of the pellet not experiencing an exact  $3^\circ\text{C}$  increase with every  $9^\circ\text{C/hr}$  increase in cartridge heater temperature. Other possibilities include uneven heating or the melting of calcium stearate.

## CHAPTER V

### CONCLUSIONS

#### Synopsis

A new “indoor” cook-off test method was designed and developed to safely heat high explosive samples at various rates to determine their critical temperature. The Petersen Group SB-IV was used as the main testing vessel and was internally lined with an aluminized heat barrier. A 400-W Watlow Firerod cartridge heater with an internal temperature sensor was the main heat source, and it was controlled using the Omega CN7823 Ramp/Soak Controller. The temperature inside the SB-IV was monitored using two separate thermocouples: one which monitored the surrounding air temperature inside the vessel, and the other to continuously monitor the temperature of the sample pellet. All data were recorded using the Omega HH378 Data Logger, recording at a rate of 1 sample/sec. This system is capable of conducting both fast and slow cook-off tests of small-scale samples for a variety of explosives and propellants.

A series of thermal cook-off tests were conducted using this setup to determine the critical temperature of pressed Nitrotriazolone (NTO) pellets for the purpose of understanding whether or not they have increased thermal stability as compared to their homogeneous counterparts. This information is supplemental to research regarding Compounded High Explosive Composite (CHEC) Technology. Four slow cook-off tests were performed in this study, reporting critical temperatures of 173.7°C, 183°C, 189.1°C and 203°C. While these values fall within reasonable range of the predicted  $T_C$  of

211.5°C, there is still some uncertainty of the actual critical temperature of the pressed pellets. The measured average critical temperature of NTO from slow cook-off is  $196 \pm 7^\circ\text{C}$ . Fast cook-off tests revealed a much more consistent temperature reading, with  $T_C$  values being recorded at 189.5°C and 188.8°C; the average critical temperature for fast cook-off was determined to be  $189 \pm 1^\circ\text{C}$ .

Nonetheless, the thermal stability of the pressed NTO pellets may still require additional testing. This result also brings about the uncertainty of the critical temperature recorded for these pellets as the reported critical temperature of NTO in literature ( $226.5 \pm 10.5^\circ\text{C}$ ) is significantly higher than those found in testing, indicating premature decomposition of the pellets during heating.

### **Challenges**

Due to high explosives being unpredictable at times, there were many safety measures that went into developing a safe “indoor” cook-off test. Preliminary designs envisioned machining a small jar-like chamber that could be placed in an oven (located inside a blast shielded cell) for heating; however, the potential risk of fragmentation caused by a pellet detonating rather than deflagrating would most likely cause severe damage to the oven. To combat this hazard, the SB-IV was selected due to its capability of withstanding extreme pressures and temperatures.

The use of the SB-IV, however, proved to have many challenges of its own. The task of heating the internal chamber of the strand burner to temperatures greater than 200°C using simply the cartridge heater revealed that the thick walls of the vessel

absorbed much of the heat generated by the heater. Wrapping the external surface of the main body with high-temperature heating tape and insulation allowed the internal air temperature to reach a maximum temperature of 105°C via the cartridge heater.

However, with this value still sizably less than the necessary 200°C, the SB-IV was then internally lined with an aluminized heat barrier capable of withstanding temperatures up to nearly 1100°C. The reason this barrier was chosen over high-temperature, silica insulation was because of how its aluminized surface is capable of reflecting over 90% of the radiant heat to which it is exposed. Using the aluminized surface with the cartridge heater, the air inside the SB-IV achieved temperatures well above 240°C.

When heating the pellet samples, test procedures initially stated the use of the ramp program of the temperature controller to automatically heat the samples at the specified rate. However, it was discovered that each of the pellets tended to heat at various rates, some achieving the subcritical, uniform temperature of 160°C faster than others. As such, the PID mode of the temperature controller was utilized to manually increase the temperature every hour, allowing for more adequate heating.

### **Recommendations**

In examining the data obtained from the slow cook-off tests, the variations in the recorded critical temperatures suggest the need for additional testing to determine a more general  $T_C$  for these specific NTO pellets. Until such data are obtained, no claims can be made about the thermal stability of CHEC arrays.

In terms of actual testing, the second setup with the horizontal strand burner was used over the initial setup with the vertical burner and the sample bolt holder due to better pellet heating. To return to the initial, more-sophisticated configuration, redesigning the sample bolt holder so that it has a smaller surface area (e.g. tapering at the neck) may reduce the amount of heat absorbed by the bolt itself, allowing for more effective heating of the pellet sample.

It was also found that the stationary air inside of the vessel tended to vary in temperature depending upon where the thermocouple was placed. For the tests included in this report, the air-measuring thermocouple and cartridge heater remained unmoved; however, the inclusion of a small, metal fan capable of withstanding extreme temperatures is worth considering to generate better heat flow. The use of a fan would keep the air within the SB-IV flowing, providing better and possibly more efficient pellet heating via forced convection.

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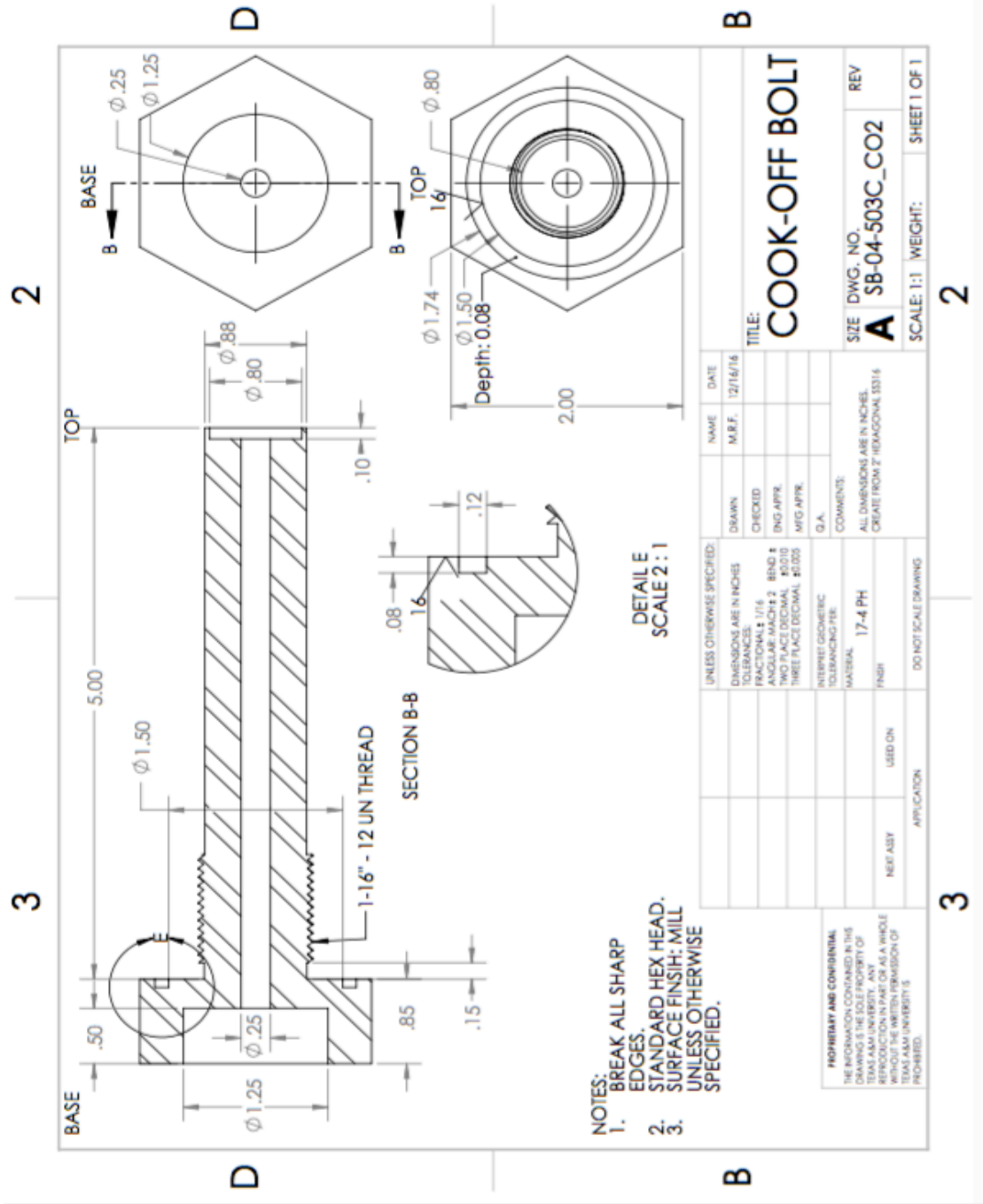
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# APPENDIX A

## SAMPLE BOLT HOLDER DRAWING



## APPENDIX B

### SYSTEM EQUIPMENT AND MATERIALS

## Cartridge/Insertion Heaters

### FIREROD Cartridge Heaters

#### Made-to-Order

#### Options

##### Passivation

During the manufacturing and handling of stainless steel, particles of iron or tool steel may embed in the sheath. If they are not removed, particles may corrode and produce rust spots. In critical sheath contact applications for the medical industry, passivation will remove free iron from the sheath. To order, specify **316L stainless steel sheath** and **passivation**.

**Note:** A minimum charge per line item applies.

##### Individually Controlled Heat Zones

Individually controlled heat zones offer the flexibility to control temperature by zones, along the length of the FIREROD heater. This is an advantage for heating requirements of certain applications, such as seal bars. This internal construction can be ordered on 5/8, 3/4 and 1 in. (16, 19 and 25 mm) diameter FIREROD heaters. To order, specify **individually controlled heat zones** and wattage and length per zone.

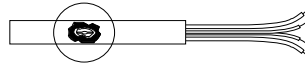
**Note:** A minimum charge per line item applies.

#### Thermocouple Types

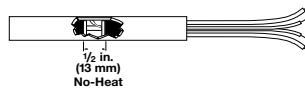
ASTM Code	Conductor Characteristics		Temperature Range °F (°C)
	Positive	Negative	
J	Iron (Magnetic) (White)	Constantan (Non-Magnetic) (Red)	0 to 1400 (-20 to 760)
K	Chromel® (Non-Magnetic) (Yellow)	Alumel® (Magnetic) (Red)	0 to 2300 (-20 to 1260)

For other ISA types, contact your Watlow representative.

##### Style A



##### Style B



##### Style C



#### Internal Thermocouple

Style A internal thermocouples can be used to evaluate heat transfer efficiency of an application. This measurement can help to cut energy costs and increase heater life. The ungrounded junction is located in the heater core to monitor the internal temperature of the heater.

The Style B internal thermocouple provides a good approximation of part temperature and is located anywhere along the length of the heater. Due to variations in production, this style may be grounded or ungrounded.

This junction is located adjacent to the inside heater sheath in the center of the heated section unless otherwise specified. A 1/2 in. (13 mm) unheated section is required.

A Style C internal thermocouple is useful in applications where material flows past the end of the heater, as in plastic molding. This grounded junction is embedded in a special end disc. Unless requested, the disc end is not mechanically sealed.

To order, specify **internal thermocouple, Style A, B or C** and **thermocouple ASTM Type J or K**.

If not specified, 12 in. (305 mm) thermocouple leads are supplied.

##### Availability

All styles are available on all diameters with the exception of 1/8 in. (3.2 mm) diameter, which is available only with Style C, and 1 in. (25 mm) which is available only with Style A and B.

# 1/16 DIN Ramp/Soak Controllers

## CN7800 Series



- ✓ Dual Display
- ✓ Autotune
- ✓ Universal Input
- ✓ 8 Ramp/Soak Programs, 8 Segments Each
- ✓ Programmable Repeat and Linking Features
- ✓ RS485 Communications
- ✓ Free Software
- ✓ 2 Alarm Standard

Monitor and control temperature or process applications with precision using the CN7800 Series controllers. The CN7800 Series provides dual LED displays for local indication of process value and setpoint value. Control methods include on/off, PID, auto-tune and manual-tune. PID control is supported with 64 ramp/soak control actions. Two additional alarm outputs are standard on the CN7800 Series. The alarm outputs can be quickly configured by using the 13 built-in alarm functions. The controller communicates easily with the built-in RS485 interface.

### Inputs

Input Types	Range
<b>K</b>	-200 to 1300°C (-328 to 2372°F)
<b>J</b>	-100 to 1200°C (-148 to 2192°F)
<b>T</b>	-200 to 400°C (-328 to 752°F)
<b>E</b>	0 to 600°C (32 to 1112°F)
<b>N</b>	-200 to 1300°C (-328 to 2372°F)
<b>R</b>	0 to 1700°C (32 to 3092°F)
<b>S</b>	0 to 1700°C (32 to 3092°F)
<b>B</b>	100 to 1800°C (212 to 3272°F)
<b>L</b>	-200 to 850°C (-328 to 1562°F)
<b>U</b>	-200 to 500°C (-328 to 932°F)
<b>Pt100 RTD</b>	-200 to 600°C (-328 to 1112°F)
<b>0 to 50 mV</b>	-999 to 9999
<b>0 to 5 V</b>	-999 to 9999
<b>0 to 10 V</b>	-999 to 9999
<b>0 to 20 mA*</b>	-999 to 9999
<b>4 to 20 mA*</b>	-999 to 9999

\* Requires external 250 Ω precision shunt resistor, OMX-R250 (sold separately). Available at [omega.com/cn7800](http://omega.com/cn7800)

Panel punches available, visit [omega.com/panelpunches](http://omega.com/panelpunches)



CN7833

### Specifications

**Inputs:** Thermocouple, RTD, DC voltage or DC current  
**Display:** Two 4-digit, 7 segment 6.35 mm H (0.25") LEDs (PV: red, SV: green)

**Resolution:** 1.0, 0.1 for thermocouples (except Types R, S and B)

**Accuracy:** ±0.25% span, ±1 least significant digit

**Supply Voltage:** 100 to 240 Vac, 50/60 Hz

**Power Consumption:** 5 VA max

**Operating Temperature:** 0 to 50°C (32 to 122°F)

**Memory Backup:** Non-volatile memory

### Control Output Ratings:

**Relay:** SPST, 5 A @ 250 Vac resistive

**Voltage Pulse:** 14V, 10 to -20% (max 40 mA)

**Current:** 4 to 20 mA

**Alarms:** SPST, 3 A @ 250 Vac resistive

**Communication:** RS485 MODBUS® A-5-11/RTU communication protocol

**Weight:** 114 g (4 oz)

**Front Bezel:** 48 mm² (1.89 in²)

**Panel Cut-Out:** 45 mm² (1.77 in²)

**Maximum Panel Thickness:** 9.50 mm (0.375")

**Panel Depth:** 80 mm (3.15")

### To Order Visit [omega.com/cn7800](http://omega.com/cn7800) for Pricing and Details

Model No.	Description
<b>CN7833</b>	Dual output, relay/relay, 2 alarms, RS485*
<b>CN7823</b>	Dual output, DC pulse/relay, 2 alarms, RS485*
<b>CN7853</b>	Dual output, 4 to 20 mA/relay, 2 alarms, RS485*

\* Free CN7-A software download available at [omega.com/cn7800](http://omega.com/cn7800)

### Accessories (Field Installable)

Model No.	Description
<b>CNQUENCHARC</b>	Noise suppression RC snubber (2 leads), 110 to 230 Vac
<b>OMX-R250</b>	250 Ω precision resistor
<b>CN7-485-USB-1</b>	RS485 to USB mini-node converter

Comes complete with operator's manual.

**Ordering Examples:** CN7823, dual-output controller, DC pulse, mechanical relay output, and RS485 communications.

CN7853, dual output 4 to 20 mA relay two alarms.

# 4-Channel Handheld Data Logger Thermometer With USB Interface

HH378



No Points

- ✓ Auto Range
- ✓ USB Interface with Windows® Software
- ✓ LED Back Light
- ✓ HOLD Function
- ✓ 4-Channel Input
- ✓ J, K, E, T Thermocouple Types
- ✓ 4-Channel Display Resolution
- ✓ 0.1°C (0.1°F) with Windows Software
- ✓ MAX/MIN/AVG Function
- ✓ Auto Shut-Off for Battery Saving
- ✓ 16,000 Records per Channel

The HH378 has a high resolution and fast analog-to-digital converter. Measurements settings and results are shown on the backlit LCD display. Data can be stored in the meter or directly saved on a computer through PC interface. Recorded data can be further processed on a computer. With the exclusive software, recording/recalling of data and programming of logging parameters can be done directly through a PC.

## Specifications

### Measurement Range:

K: -200 to 1372°C (-328 to 2501°F)  
J: -150 to 1000°C (-238 to 1832°F)  
E: -150 to 750°C (-238 to 1382°F)  
T: -180 to 400°C (-292 to 752°F)

**Input Protection:** 250 Vac

**Resolution:** 0.1°C/°F <1000, 1°C/°F ≥1000

**Accuracy:** ±(0.1% of reading + 0.7°C)  
±(0.1% of reading + 1.4°F)

**Below -100°C (-148°F):**

±(0.4% of reading + 0.7°C)

±(0.4% of reading + 1.4°F)

### Temperature Coefficient:

0.01% of reading + 0.05°C per °C  
(<18°C or >28°C)

**Sample Rate:** 1 time per second

**Battery Type:** 9V battery (included)

**Battery Lifetime:** Approximately 30 hours (alkaline battery)

**AC Adaptor:** 9 Vdc (8 to 10 Vdc maximum)

**Operating Temperature:** -10 to 50°C  
(14 to 122°F)

**Operating Humidity:** 10 to 90% RH  
(no condensing)

**Storage Temperature:** -40 to 60°C  
(-40 to 140°F)

**Storage Humidity:** 10 to 75% RH

### Dimensions:

196 H x 65 W x 36 mm D  
(7.72 x 2.56 x 1.42")

### Approximate Weight:

310 g (10.93 oz)

**System Required:** Windows NT 4.0/  
NT2000/XP/Vista/Windows 7



OMEGACARE™ extended warranty program is available for models shown on this page. Ask your sales representative for full details when placing an order. OMEGACARE™ covers parts, labor and equivalent loaners.

## Free Thermocouples Included!

This model includes 4 free 1 m (40") Type K insulated beaded wire thermocouples with subminiature connector and wire spool caddy (1 per channel).  
**Order a Spare! Model No. SC-GG-K-30-36**



HH378 shown smaller than actual size.

## To Order

Model No.	Description
HH378	4-channel handheld data logger thermometer

## Accessories (Field Installable)

Model No.	Description
HH300-ADAPTOR	Power adaptor (115 Vac)
HH300A-CABLE-USB	Spare USB interface cable

Comes complete with 4 beaded wire Type K thermocouples, 9V battery, hard carrying case, software, USB cable, NIST certificate, and operator's manual.

**Ordering Example:** HH378, 4-channel data logger thermometer. OCW-3, OMEGACARE™ extends standard 1-year warranty to a total of 4 years.

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# **ALUMINIZED HEAT BARRIER™ ADHESIVE BACKED**

- Reflects radiant heat
- Withstands radiant heat up to 2,000° F
- Installs virtually anywhere
- Protect vital components



**2000**  
DEGREES  
RADIANT HEAT REFLECTION

REFLECTS OVER  
**90%**  
OF RADIANT HEAT

